Atlantic Richfield Company

1290029

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January 30, 2014

Mr. Steven Way
On-Scene Coordinator
Emergency Response Program (8EPR-SA)
U.S. EPA, Region 8
1595 Wynkoop Street
Denver, CO 80202-1129

RE: Supplemental Sample Analysis Results
St. Louis Tunnel Discharge Constructed Wetland Pilot Scale Test Completion Report Addendum
Rico-Argentine Mine Site – Rico Tunnels, Operable Unit OU01
Dolores County, Colorado

Dear Mr. Way:

On behalf of Atlantic Richfield Company (Atlantic Richfield), please find enclosed the *St. Louis Tunnel Discharge Constructed Wetland Pilot Scale Test Completion Report Addendum: Supplemental Sample Analysis Results* (Report), which documents the results of supplemental sample collection and scanning electron microscope (SEM) analysis associated with the St. Louis Tunnel Discharge Constructed Wetland (pilot-scale wetland) Pilot Scale Test (pilot test). This Report is provided as an addendum *St. Louis Tunnel Discharge Constructed Wetland Pilot Scale Test Completion Report* (Completion Report), which was submitted in November 2013 pursuant to the requirements in Task F – Water Treatment System Analysis and Design / Subtask F2 – Treatment System Conceptual Designs and Additional Investigations of the Remedial Action Work Plan accompanying the Unilateral Administrative Order for Removal Action, Rico-Argentine Site, Dolores County, United States Environmental Protection Agency, Region 8, (U.S. EPA), dated March 9, 2011 (Docket No. CERCLA-08-2011-0005).

This Report describes the sample locations and methods and presents analytical results (i.e., elemental composition and visual observations) that were unavailable for inclusion in the Completion Report. Samples were collected downgradient of the pilot-scale wetland discharge point in September 2013 and submitted to the Dedman College of Humanities & Sciences Department of Earth Sciences SEM Laboratory at Southern Methodist University (SMU) in Dallas, Texas, for SEM analysis. As stated in the Completion Report transmittal letter (November 4, 2013) and indicated in responses to the U.S. EPA's comments on the *Constructed Wetland Demonstration Treatability Study Work Plan* (see page 4 of the Response to Comments letter dated October 9, 2013, regarding Section 4.3), these samples were collected to verify the composition of material that was observed near the pilot-scale wetland effluent. The results reported herein provide a better understanding of the biogeochemical processes that occurred in the pilot-scale wetland and the effluent mixing zone within Pond 9.

If you have any questions regarding this Report, please feel free to contact me at (714) 228-6770 or via e-mail at Anthony.Brown@bp.com.

Sincerely,

Tony Brown
Project Manager Mining
Atlantic Richfield Company

Enclosure: *St. Louis Tunnel Discharge Constructed Wetland Pilot Scale Test Completion Report Addendum: Supplemental Sample Analysis Results*

cc: Ronald Halsey, Atlantic Richfield Company (via e-mail)
Terry Moore, Atlantic Richfield Company (via e-mail)
Sheila D'Cruz, Atlantic Richfield Company (via e-mail)
Cord Harris, Atlantic Richfield Company (via e-mail)
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Chris Sanchez, Anderson Engineering Company, Inc. (via e-mail)
Dave McCarthy, Copper Environmental Consulting, LLC (via e-mail)
Jan Christner, Weston Solutions, Inc. (via e-mail)

ST. LOUIS TUNNEL DISCHARGE
CONSTRUCTED WETLAND PILOT SCALE TEST
COMPLETION REPORT
ADDENDUM:

SUPPLEMENTAL SAMPLE ANALYSIS RESULTS

Rico-Argentine Mine Site – Rico Tunnels
Operable Unit OU01
Dolores County, Colorado

Prepared for:
Atlantic Richfield
La Palma, California

Prepared by:
AMEC Environment & Infrastructure, Inc.
Rancho Cordova, California

January 2014

Project No. SA11161340.5

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FIGURE

ATTACHMENTS

ABBREVIATIONS

Al	aluminum
AMEC	AMEC Environment & Infrastructure, Inc.
Atlantic Richfield	Atlantic Richfield Company
C	carbon
Ca	calcium
$CaSO_4$	gypsum
Completion Report	St. Louis Tunnel Discharge Constructed Wetland Pilot Scale Test Completion Report
EDS	energy-dispersive spectrometer
Fe	iron
O	oxygen
pilot-scale wetland	St. Louis Tunnel Discharge Constructed Wetland
pilot test	St. Louis Tunnel Discharge Constructed Wetland Pilot Scale Test
RAWP	Removal Action Work Plan
Report	St. Louis Tunnel Discharge Constructed Wetland Pilot Scale Test Completion Report Addendum: Supplemental Sample Analysis Results
S	sulfur
SAP	Sampling and Analysis Plan
SEM	scanning electron microscope
Si	silicon
site	Rico-Argentine Mine Site – Rico Tunnels, Operable Unit OU01, Dolores County, Colorado
SMU	Dedman College of Humanities & Sciences Department of Earth Sciences SEM Laboratory at Southern Methodist University
sonde	YSI 6920 multi-parameter sonde
U.S. EPA	United States Environmental Protection Agency
Zn	zinc
µm	micron, micrometer
%	percent

ST. LOUIS TUNNEL DISCHARGE
CONSTRUCTED WETLAND PILOT SCALE TEST
COMPLETION REPORT ADDENDUM:
SUPPLEMENTAL SAMPLE ANALYSIS RESULTS

Rico-Argentine Mine Site – Rico Tunnels
Operable Unit OU01
Dolores County, Colorado

1.0 INTRODUCTION

AMEC Environment & Infrastructure, Inc. (AMEC), on behalf of Atlantic Richfield Company (Atlantic Richfield), has prepared this *St. Louis Tunnel Discharge Constructed Wetland Pilot Scale Test Completion Report Addendum: Supplemental Sample Analysis Results* (Report) to document the results of supplemental sample collection and analysis associated with the St. Louis Tunnel Discharge Constructed Wetland (pilot-scale wetland) Pilot Scale Test (pilot test). This Report is provided as an addendum to the *St. Louis Tunnel Discharge Constructed Wetland Pilot Scale Test Completion Report* (Completion Report; AMEC, 2013), which was prepared pursuant to Task F Subtask F2 of the *Removal Action Work Plan* (RAWP) attached to the *Unilateral Administrative Order* issued to Atlantic Richfield by the United States Environmental Protection Agency (U.S. EPA) on March 9, 2011 (Docket No. 08-2011-0005; U.S. EPA, 2011) for the Rico-Argentine Mine Site – Rico Tunnels, Operable Unit OU01, Dolores County, Colorado (site).

Throughout the pilot test, black and white surface deposits were observed near the pilot-scale wetland effluent point. The inside of the pilot-scale wetland effluent pipe and the rocks near the effluent discharge point were coated with creamy white, filamentous deposits indicative of the presence of biofilm and elemental sulfur. Black deposits that accumulated on the ground surface below the effluent discharge point and black colloidal material in the Pond 9 mixing zone (i.e., where the pilot-scale wetland effluent contacts Pond 9 waters, downgradient of the pilot-scale wetland effluent discharge point) were presumed to be metal sulfides. Greyish suspended colloids in the Pond 9 mixing zone were presumed to be elemental sulfur.

On September 19, 2013, water and sediment samples were collected near the pilot-scale wetland effluent discharge pipe and submitted to an analytical laboratory for scanning electron microscope (SEM) analysis to verify the elemental composition of materials. Three samples were collected consisting of material that had accumulated in and below the pilot-scale wetland effluent discharge pipe. These samples were presumed to consist primarily of biofilm, elemental sulfur, and metal sulfides. Additionally, two water samples from the effluent mixing zone in Pond 9 were filtered, and the particulate material that was retained on the filters was submitted for analysis.

The results of SEM analysis (i.e., elemental composition and visual observations) are presented in this Report. These results were used to evaluate the chemical composition of the materials and better understand the biogeochemical processes that occurred in the pilot-scale wetland and the effluent mixing zone within Pond 9. Additional treatability study results, as well as details of the wetland pilot test construction and operation, are presented in the Completion Report (AMEC, 2013).

2.0 SAMPLE COLLECTION, HANDLING, AND ANALYSIS

The following sections describe the sample locations, sampling methods, and analytical methods employed for the SEM samples obtained near the pilot-scale wetland effluent.

2.1 SAMPLE LOCATIONS AND METHODOLOGY

On September 19, 2013, five samples were collected near the pilot-scale wetland effluent discharge pipe and submitted to an analytical laboratory for SEM analysis. Photographic documentation of the sampling event is provided as Attachment A. The locations and collection methods for each sample are described below.

2.1.1 Laboratory Sample #1

Laboratory Sample #1 (Field Sample POND9FILTRATE13091901) was collected with a peristaltic pump to filter water from the mixing zone in Pond 9 through a flat 0.45-micron (µm) filter. The pump intake was located within Pond 9, about five feet west of the pilot-scale wetland discharge point (Photographs 1 and 2 of Attachment A), in an area of the pond where suspended black colloids had been observed visually. This sample was expected to consist of suspended particles, including colloidal metal sulfides.

2.1.2 Laboratory Sample #2

Laboratory Sample #2 (Field Sample POND9FILTRATE13091902) was similarly collected by filtering water from the mixing zone in Pond 9 through a flat 0.45-µm filter. The pump intake was located within Pond 9, about eight feet west of the pilot-scale wetland discharge point (Photographs 3 through 7 of Attachment A), in an area of the pond where suspended grayish colloids had been observed visually. This sample was expected to consist of suspended particles, including colloidal metal sulfides.

2.1.3 Laboratory Sample #3

Laboratory Sample #3 (Field Sample BIOFILMSTANDPIPE130919) consisted of material that had accumulated on the YSI 6920 multi-parameter sonde (sonde) that was deployed in the standpipe at the pilot-scale wetland outlet. The material was scraped off the sonde and onto a coffee filter, which had been suspended over a glass jar to eliminate some of the moisture (Photographs 8 through 10 of Attachment A). After a short drying period, the coffee filter was wrapped around the sampled media and placed inside the glass jar for submittal to the analytical laboratory. This sample was thought to consist of biofilm and/or elemental sulfur.

2.1.4 Laboratory Sample #4

Laboratory Sample #4 (Field Sample BIOFILMROCK130919) consisted of material that had accumulated on the surface of the rocks immediately downgradient of the pilot-scale wetland discharge point. The material was scraped off several rocks and onto a coffee filter, which had been suspended over a glass jar to eliminate some of the moisture (Photographs 11 through 13 of Attachment A). After a short drying period, the coffee filter was wrapped around the sampled media and placed inside the glass jar for submittal to the analytical laboratory. This sample was thought to consist primarily of elemental sulfur.

2.1.5 Laboratory Sample #5

Laboratory Sample #5 (Field Sample BLACKSEDIMENT130919) consisted of black material that had accumulated on subsurface rocks beneath the sulfur/biofilm covered rocks immediately downgradient of the pilot-scale wetland discharge point. The material was scraped off several rocks and onto a coffee filter, which had been suspended over a glass jar to eliminate some of the moisture (Photographs 14 and 15 of Attachment A). After a short drying period, the coffee filter was wrapped around the sampled media and placed inside the glass jar for submittal to the analytical laboratory. This sample was thought to consist primarily of metal sulfides due to the black appearance of the sample.

2.2 SAMPLE PREPARATION AND ANALYSIS

The five samples were shipped on ice, with no preservation, under chain of custody, to the Dedman College of Humanities & Sciences Department of Earth Sciences SEM Laboratory at Southern Methodist University (SMU) in Dallas, Texas, for delivery on September 24, 2013. Samples were prepared for SEM analysis by air drying and mounting for examination. Some oxidation of sample components (particularly metal sulfides) may have occurred during sample handling and preparation. Samples were not coated with carbon, gold, or other conductive material during preparation. Analyses were completed using a Leo-Zeiss 1450VPSE variable pressure electron microscope equipped with an EDAX Genesis 4000 XMS SYSTEM 60 energy-dispersive spectrometer (EDS). Instrumentation specifications are described on the laboratory's web page (SMU, 2013).

Each sample was subjected to multiple analyses. The initial analysis of each sample quantified the elemental composition of a relatively broad area of the sample, while additional analyses focused on specific locations or features within the sample field using various magnifications.

3.0 RESULTS AND DISCUSSION

The following presents and interprets the analytical results for the SEM samples obtained near the pilot-scale wetland effluent. Results for each analysis, as provided by SMU, are provided in Attachment B and include the EDS spectrum, tables of elemental composition, and a backscatter mode photograph of the sample field. For Laboratory Sample #1, an additional high resolution photograph using the primary electron beam is provided as Figure 1.

3.1 LABORATORY SAMPLE #1

Laboratory Sample #1 consisted of solid material (primarily black), obtained from the mixing zone of the pilot-scale wetland effluent in Pond 9, that was retained on a 0.45-μm filter. This sample was analyzed 12 times by SEM. Highlights of visual observations and elemental analyses are summarized below.

- Sample1_01 – This broad characterization of Laboratory Sample #1 shows the elemental composition of the entire sample within the red box (in this case the entire frame). The area had substantial mineral and organic material (about 20 percent [%] carbon [C] by weight) present as colloids retained on the filter. The sample has a substantial amount of iron (Fe), indicating the presence of Fe-containing minerals, possibly Fe sulfide. Zinc (Zn) also may be present as zinc sulfide. The presence of sulfur (S) at about 11% by weight indicates the presence of particulate metal sulfides and/or elemental S associated with this sample. The presence of silicon (Si) is an indicator of diatoms and aluminosilicate minerals, which are confirmed in photographs of other Sample #1 locations (Sample1_04 through Sample1_12 in Attachment B), as well as in Figure 1.

- Sample1_02 – The elemental composition in the red box is a subsection of Sample 1_01 and was very similar in composition. Presence of Si (about 9% by weight) may indicate the presence of diatoms (and aluminosilicate sediment particles) on the filter, and the presence of Fe and Zn likely indicates that these metals were contained within or adsorbed to colloids. Magnification was insufficient for visual analysis of individual particles.

- Sample1_03 – Similar chemical composition to that of Sample1_01 and Sample1_02. The long linear feature did not cause an appreciable difference in chemical composition.

- Sample1_04 – Higher magnification, with crosshair focused on a diatom filtered from pond water. The presence of C, oxygen (O), and Si, as well as distinctive morphology, indicates that this is a 30 μm-long diatom. As with previous samples, the presence of Fe, Zn, and S likely indicates colloids containing these metals.

- Sample1_05 – Crosshair focused on a diatom, as evidenced with high Si content (about 29% by weight). Other detected elements may be in the vicinity of the focus point and close enough to give a signature during analysis. The presence of S (about 5.7% by weight) may indicate sulfate or sulfide, possibly associated with Zn and/or Fe. This sample was collected from an area of Pond 9 that appeared black, likely due to suspended metal sulfide particles. Thus, when the sample was collected, S may have been present in sulfide form and associated with Fe and Zn, but sulfide may have oxidized to sulfate during storage, transit, preparation, and analysis.

- Sample1_06 – Crosshair focused on what appears to be another diatom with different morphology than those of Sample1_04 and Sample1_05, as evidenced by high Si and presence of C. Alternatively, the high Si could indicate the presence of a silt-sized quartz particle.

- Sample1_07 – Due to relatively high Fe concentration, this observation appears to be and aggregate containing Fe oxides, with some S and Zn. These materials may have originated as colloidal Fe or Zn sulfides discharged from the wetland and oxidized in Pond 9. Both of these sulfides, particularly when biogenic, are reactive and tend to oxidize rapidly.

- Sample1_08 – The crosshair appears to be focused on some sort of large organic particle, possibly plant material, as indicated by high carbon content. Smaller particles also excited by the electron beam may include sulfides of Fe and Zn.

- Sample1_09 – This appears to be another diatom or a different type of algae, with substantial C and Si. The presence of Zn and Fe may indicate adherence of these metals to the cell wall or may be due to other materials in the vicinity of the focus point (electron beam) that yield a signature during analysis.

- Sample1_10 – The particle in this location had high S concentration (70% by weight), indicating that the focus is on an elemental sulfur granule. Other particles of similar size, shape, and brightness are observed in the field of view, strongly suggesting the presence of other S granules in this view. Based on the 20-μm scale, the diameter of many of these granules is about 2-5 μm, and are clay or small silt-sized particles. Darker material in this view may consist of fine Fe- and Zn-rich particles (likely sulfides), which may have been discharged from the wetland as colloidal metal sulfides or may have been formed in the pond by reaction of discharged sulfides with metals in pond water.

- Sample1_11 – Same field of view with a different particle of similar size and morphology as a check of consistency. The particle again shows extremely high concentrations of S and is likely an elemental S particle. Figure 1 shows the same field of view but utilized the primary electron beam to obtain a higher resolution photograph. Figure 1 shows a diatom, elemental S (including the rhombic structure of the particle that was analyzed for Sample1_11), metal sulfides, and possibly plant material. Low Si with relatively high C and O indicate that this location may contain biofilm.

- Sample1_12 – This location has high S (about 22% by weight) and calcium (Ca; about 21% by weight), possibly indicating the presence of a gypsum ($CaSO_4$) particle or gypsum precipitated on the surface during the drying preparation. Surrounding materials (also excited by the electron beam) include Fe- and Zn-containing minerals. Low Si with relatively high C and O indicate that this location likely contains biofilm.

In summary, solid material that was retained on the 0.45-µm filter for Laboratory Sample #1 included a diverse array of particles. SEM results indicate that this sample included diatoms and other biomass, as well as metal sulfides and elemental sulfur particles with a diameter of about 2-5 µm. Sulfur particles may have been formed by oxidation of sulfides in the wetland or in the Pond 9 mixing zone.

3.2 LABORATORY SAMPLE #2

Laboratory Sample #2 consisted of solid material (primarily black/gray) from the mixing zone of the wetland effluent in Pond 9 that was retained on a 0.45-µm filter. The sample tubing intake was placed about eight feet west of the wetland discharge location, from an area of the pond with a lighter milky white/grey color than Laboratory Sample #1. This sample was analyzed five times by SEM. Highlights of visual observations and elemental analyses of Laboratory Sample #2 are summarized below.

- Sample2_01 – This sample had elemental S, which stands out as bright particles as with Sample1. This sample also had substantial biomass, as indicated by the relatively high C content (about 49% by weight). Although unclear from these results, biomass was being discharged to Pond 9 with the wetland effluent or may be material indigenous to Pond 9. The low Si content indicates that the biomass is not algae.

- Sample2_02 – The particle at this location had relatively high S, Ca, and O. The material at the point of focus is substantially larger (about 20 µm by 10 µm) than other features in this view. This material may be gypsum ($CaSO_4$) crystals that formed when samples were dried. Oxidation of sulfides may have been the source of some of the sulfate. The relatively high C may also indicate the presence of biofilm or organic material in this sample, as indicated in Sample2_01.

- Sample2_03 – This location in the same field of view shows discrete 1 to 5-µm particles, which are likely elemental S (about 59% by weight). The high C (about 29% by weight) may be organic material from the wetland or from biomass produced in the existing pond.

- Sample2_04 – This location was not a discrete particle, but fine-grained organic material adhering to the filter with relatively high C concentration. The presence of S may indicate the presence of some Fe and Zn sulfides in the vicinity. No diatoms were present in this sample due to low Si content and lack of visual evidence.

- Sample2_05 – This location was primarily elemental S, reflecting the large number of these particles throughout the sample.

In summary, solid material that was retained on the filter for Laboratory Sample #2 generally had fine silt-sized granules with high sulfur content, indicating the presence of elemental S particles. This sample was collected from the area of Pond 9 with a grayish white color, likely indicating the presence of suspended elemental sulfur particles in this area of the pond. Sulfur particles may have been discharged from the wetland or formed by oxidation of sulfides in Pond 9. Very fine-grained organic matter and likely some Fe and Zn sulfides were also common in the sample.

3.3 LABORATORY SAMPLE #3

Laboratory Sample #3 consisted of grayish material (thought to be biofilm with elemental sulfur) that had accumulated on the sonde that was deployed in the standpipe at the wetland outlet. The material was analyzed five times by SEM; visual observations and elemental analyses are summarized below.

- Sample3_01 – This lower-magnification sample location had relatively high O, C, and S content, strongly suggesting the presence of biofilm and elemental S. The small amount of Ca may indicate presence of $CaCO_3$. This sample had no Fe or Zn detected, indicating that particles with these metals were not formed or retained at this location.

- Sample3_02 – This sample had high S content (88% by weight), with some C and O, indicating that this location was biofilm with associated S. Filamentous material observed in the photograph consist of cemented elemental S in the form of the stranded microorganisms that oxidize sulfide is to elemental S within the biofilm.

- Sample3_03 – Very similar to Sample3_02, with higher magnification. Uniform, small S granules (about 1 μm diameter) appear to be interwoven into biofilm strands. Small S granules. The filamentous particle under the cross hairs was analyzed to determine if it was organic, but it was primarily elemental S.

- Sample3_04 – Another particle consistent with Sample3_03.

- Sample3_05 – Appears to show formation of gypsum ($CaSO_4$), as the Ca and C contents are not high enough to indicate calcium carbonate ($CaCO_3$); the morphology also appears to be consistent with gypsum. The gypsum crust at this location may be an artifact of drying during sample preparation. The presence of C and O may also indicate that this location contains biofilm or other organic material.

Laboratory Sample #3 appears to be biofilm with elemental sulfur particles. Formation of gypsum indicates some sample oxidation prior to analysis.

3.4 LABORATORY SAMPLE #4

Laboratory Sample #4 consisted of material (thought to be biofilm and/or elemental sulfur) that had accumulated on the rocks below the system discharge point. The material was analyzed twice, and results are summarized below.

- Sample4_01 – Results were similar to Laboratory Sample #3, as expected. The high S content (nearly 90% by weight) along with some C (about 8.6% by weight) strongly indicates that this sample is biogenic elemental sulfur associated.
- Sample4_02 – The larger particles (shown in the left of the photograph) are probably S particles that were formed in the wetland and were washed from the system. The smaller bright particles (0.5 – 1 μm in diameter) may be bacteria (indicated by consistent size) with associated S granules that may have been formed in a biofilm as sulfide was oxidized to S.

Laboratory Sample #4 appears to consist primarily of biogenic elemental sulfur particles, either discharged from the system or formed in-place by oxidation of sulfide with the biofilm on these rocks.

3.5 LABORATORY SAMPLE #5

Laboratory Sample #5 consisted of material that had accumulated on rock surfaces beneath the sulfur/biofilm covered rocks at the wetland outlet. The material was thought to consist of metal sulfides and other particulates that had accumulated on the rocks directly beneath the effluent discharge point. The material was analyzed six times, and results are summarized below.

- Sample5_01 – This sample appears to include amorphous metal sulfides, formed by interaction of discharged sulfide with dissolved metals in the underlying soil. Fe sulfides probably dominate, as evidenced by the relatively high Fe content (nearly 24% by weight). The presence of aluminum, potassium, and Si may indicate that some clay minerals or other soil particles were mixed into this sample.
- Sample5_02 – The aggregate particle that was analyzed appears to have a similar composition to Sample5_01. This may be a cemented aggregate particle with associated Fe sulfide.

- Sample5_03 – This location appears to have substantial Fe sulfides, and possibly Fe oxides. Reduced water discharged from the system may have resulted in reductive dissolution of underlying minerals, which further reacted to form oxides and sulfides.

- Sample5_04 – This location appears to be an aggregate particle (with a diameter of about 15 µm) composed of fine particles adhering together. The relatively high aluminum (Al) and Si content indicates the presence of clay particles. Amorphous Fe and Zn sulfides may be present, possibly precipitated at the surface of the clay particles.

- Sample5_05 – This location appears to be a particle that is similar to the particle in Sample5_04, although slightly larger (diameter of 20 to 30 µm). As with Sample5_04, the particle likely includes clays (indicated by the presence of Al and Si) with associated Fe and Zn sulfides (or sulfates). Additional S may be present as elemental S particles. This location has substantial C (nearly 23% by weight), which may indicate organic material (possibly humic material from wetland matrix decomposition) associated with the clay particles.

- Sample5_06 – This location had similar results to Sample5_05, but more Al and Si reflecting more clay mineral content.

In summary, Laboratory Sample #5 appears to have Fe and Zn sulfides that were discharged from the system or that formed by reactions with metals in the sediment. The sample also contains clay, organic matter, and other soil minerals from the sediment.

4.0 CONCLUSIONS

Visual observations and elemental analyses of these samples provide evidence for several biogeochemical processes occurring near the pilot-scale wetland effluent.

- The presence of elemental S particles was clearly demonstrated in four of the five samples (all samples except Laboratory Sample #5). These results demonstrate that elemental sulfur is present in the pilot-scale wetland effluent and/or upon oxidation in the Pond 9 mixing zone. Elemental S may be formed biogenically in some areas and chemically in others, both from oxidation of sulfides.

- The pilot-scale wetland effluent (standpipe sample, Laboratory Sample #3) clearly had elemental sulfur associated with biofilm, indicating a robust population of sulfide-oxidizing microorganisms. The biological scale adheres well to surfaces.

- Several samples had evidence for iron and zinc sulfides, which may have been particles discharged from the pilot-scale wetland or formed during reactions of discharged sulfides with metals already in the pond water.

- The occurrence of diatoms in Laboratory Sample #1 indicates that these aquatic organisms are present in the Pond 9 mixing zone.

Although some metal sulfides may have oxidized before analysis, the results indicate that the expected processes were occurring, i.e., biogenic production of elemental sulfur via sulfide oxidation and formation of metal sulfides in the pilot-scale wetland and Pond 9.

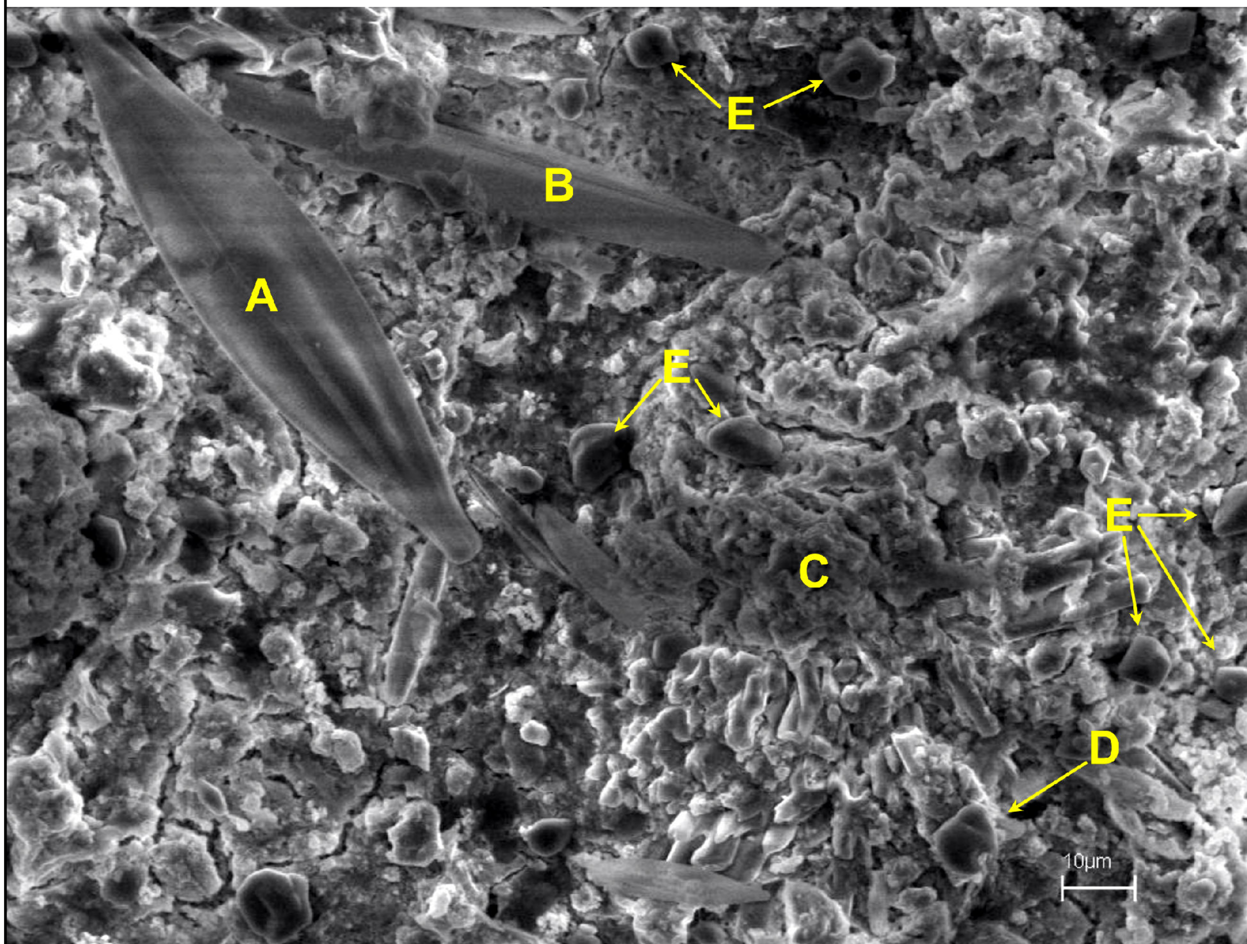
5.0 REFERENCES

AMEC, 2013, St. Louis Tunnel Discharge Constructed Wetland Pilot Scale Test Completion Report, Rico-Argentine Mine Site – Rico Tunnels, Operable Unit OU01, Dolores County, Colorado, prepared for Atlantic Richfield, submitted to U.S. EPA, Region 8, November 4.

SMU, 2014, Scanning Electron Microscope Laboratory, Southern Methodist University, Dedman College of Humanities & Sciences, available online at http://www.smu.edu/Dedman/Academics/Departments/EarthSciences/Research/Facilities/VPSEM%20Lab, accessed 20 January.

U.S. EPA, 2012, Removal Action Work Plan, Rico-Argentine Mine Site – Rico Tunnels, Operable Unit OU01 Rico, Colorado, U.S. Environmental Protection Agency, Region 8, for Atlantic Richfield Company, 9 March.

FIGURES



Note:
View is similar to that shown for Sample1_10 through Sample 1_12 (Attachment 3), with higher magnification and using primary electron imaging rather than backscatter mode. Notable features: (A) diatom; (B) plant material; (C) metal sulfide solids; (D) elemental sulfur particle (as identified on Sample1_11); and (E) elemental sulfur particles.

PRIMARY ELECTRON BEAM IMAGE OF SAMPLE 1
(Field Sample POND9FILTRATE13091901)
Rico-Argentine Mine Site
Dolores County, Colorado

By: dpv	Date: 01/30/2014	Project No. SA11161300
amec		Figure 1

ATTACHMENT A

Photographic Documentation of Field Sampling Event

ATTACHMENT A

Photographic Documentation of Field Sampling Event
Rico-Argentine Mine Site
Dolores County, Colorado

Photograph 1 Sampling location for Laboratory Sample #1 from Pond 9.

Laboratory Sample #1
Field Sample ID: **POND9FILTRATE13091901**
Colloidal materials (though to be metal sulfides due to black color of suspended materials) from the wetland effluent mixing zone in Pond 9, approximately 5 feet west of the wetland discharge location. Sample was collected with a peristaltic pump and 0.45 micron flat filter.

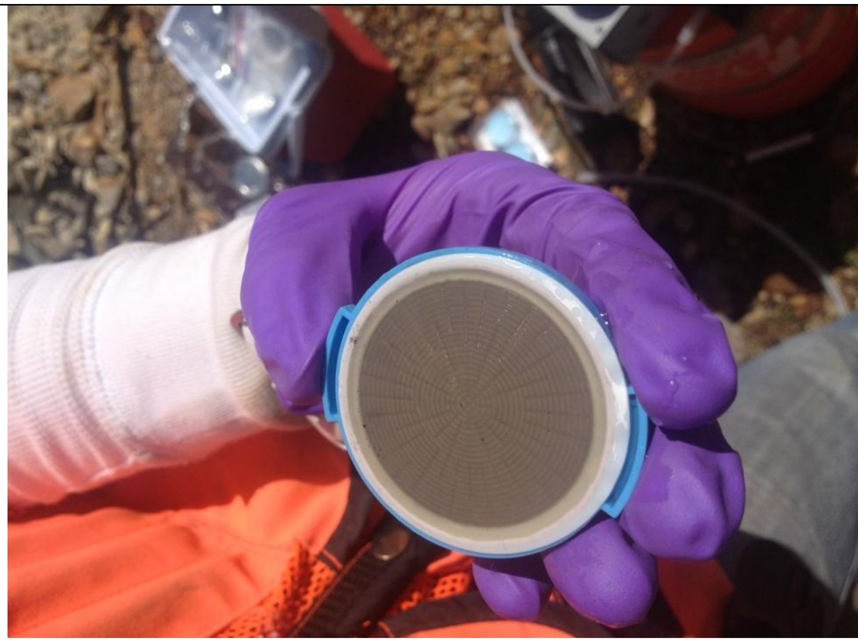
Photograph 2 Filtration apparatus.

Laboratory Sample #1 (continued)
Field Sample ID: **POND9FILTRATE13091901**
Colloidal materials from the wetland effluent mixing zone in Pond 9, approximately 5 feet west of the wetland discharge location. Sample was collected with a peristaltic pump and 0.45 micron flat filter. Filter with retained black material was submitted for analysis.

ATTACHMENT A
Photographic Documentation of Field Sampling Event
Rico-Argentine Mine Site
Dolores County, Colorado

Photograph 3 Sampling location for Laboratory Sample #2 from Pond 9.

Laboratory Sample #2
Field Sample ID: **POND9FILTRATE13091902**
Colloidal materials (though to be elemental sulfur due to white/grey color of suspended materials) from the wetland effluent mixing zone in Pond 9, approximately 8 feet west of the wetland discharge location. Sample was collected with a peristaltic pump and 0.45 micron flat filter.

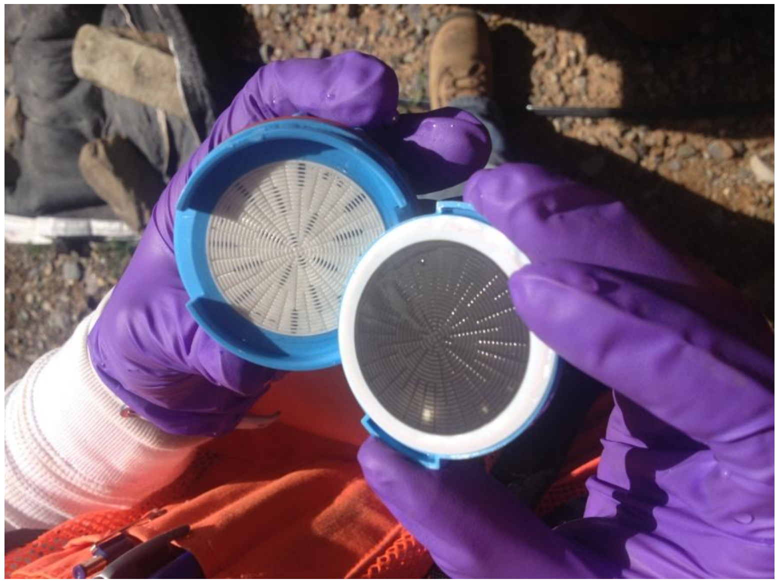


Photograph 4 Laboratory Sample #2, filter #1 with retained material.

Laboratory Sample #2 (continued)
Field Sample ID: **POND9FILTRATE13091902**
Colloidal materials from the wetland effluent mixing zone in Pond 9, approximately 8 feet west of the wetland discharge location. Sample was collected with a peristaltic pump and 0.45 micron flat filter. Four filters with retained grayish material were submitted for analysis.

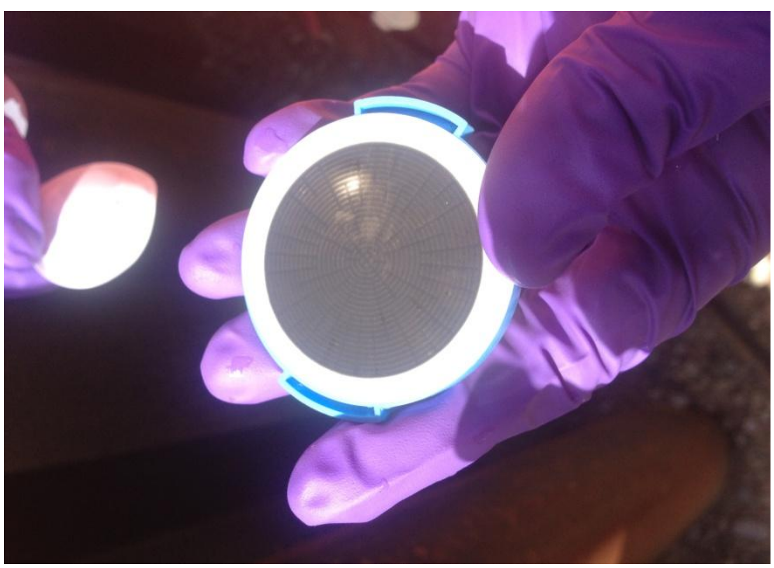
Photograph 5 Laboratory Sample #2, filter #2 with retained material.

ATTACHMENT A
Photographic Documentation of Field Sampling Event
Rico-Argentine Mine Site
Dolores County, Colorado



Photograph 6 Laboratory Sample #2, filter #3 with retained material.

Laboratory Sample #2 (continued)
Field Sample ID: **POND9FILTRATE13091902**
Colloidal materials from the wetland effluent mixing zone in Pond 9, approximately 8 feet west of the wetland discharge location. Sample was collected with a peristaltic pump and 0.45 micron flat filter. Four filters with retained grayish material were submitted for analysis.



Photograph 7 Laboratory Sample #2, filter #4 with retained material.

ATTACHMENT A

Photographic Documentation of Field Sampling Event
Rico-Argentine Mine Site
Dolores County, Colorado

Photograph 8 Standpipe at subsurface flow wetland effluent.

Laboratory Sample #3
Field Sample ID: **BIOFILMSTANDPIPE130919**
Accumulated material (possibly elemental sulfur and/or biofilm) was sampled from the surface of the sonde deployed in the wetland effluent standpipe.

Photograph 9 Sample collected from material accumulated on the sonde sensor protective cover

ATTACHMENT A
Photographic Documentation of Field Sampling Event
Rico-Argentine Mine Site
Dolores County, Colorado

Photograph 10 Sample placed onto a mason jar fitted with a coffee filter to reduce moisure content of the sample.

Laboratory Sample #3 (continued)
Field Sample ID: **BIOFILMSTANDPIPE130919**
Accumulated material (possibly elemental sulfur and/or biofilm) was sampled from the surface of the sonde deployed in the wetland effluent standpipe.

ATTACHMENT A

Photographic Documentation of Field Sampling Event
Rico-Argentine Mine Site
Dolores County, Colorado

Photograph 11 Grayish material (possibly elemental sulfur and/or biofilm) covering the rocks below wetland effluent discharge point.

Laboratory Sample #4
Field Sample ID: **BIOFILMROCK130919**
Accumulated material (possibly elemental sulfur and/or biofilm) was sampled from the surface of the rocks below the wetland effluent discharge point.

Photograph 12 Removing coating from rocks.

ATTACHMENT A
Photographic Documentation of Field Sampling Event
Rico-Argentine Mine Site
Dolores County, Colorado

Photograph 13 Sample material removed from rocks and placed on coffee filter to reduce moisture content.

Laboratory Sample #4 (continued)
Field Sample ID: **BIOFILMROCK130919**
Accumulated material (possibly elemental sulfur and/or biofilm) was sampled from the surface of the rocks below the wetland effluent discharge point.

ATTACHMENT A

Photographic Documentation of Field Sampling Event
Rico-Argentine Mine Site
Dolores County, Colorado

Photograph 14 Collecting sediment sample, after displacing upper rocks.

Laboratory Sample #5
Field Sample ID: **BLACKSEDIMENT130919**
Black sediment (presumed to be metal sulfides) was sampled from beneath the surface rocks that were coated with sulfur and/or biofilm (Laboratory Sample #4). Material was beneath wetland effluent discharge point.

Photograph 15 Black sediment material, placed on coffee filter to reduce moisutre content.

ATTACHMENT B

Laboratory Reports from SMU SEM Laboratory

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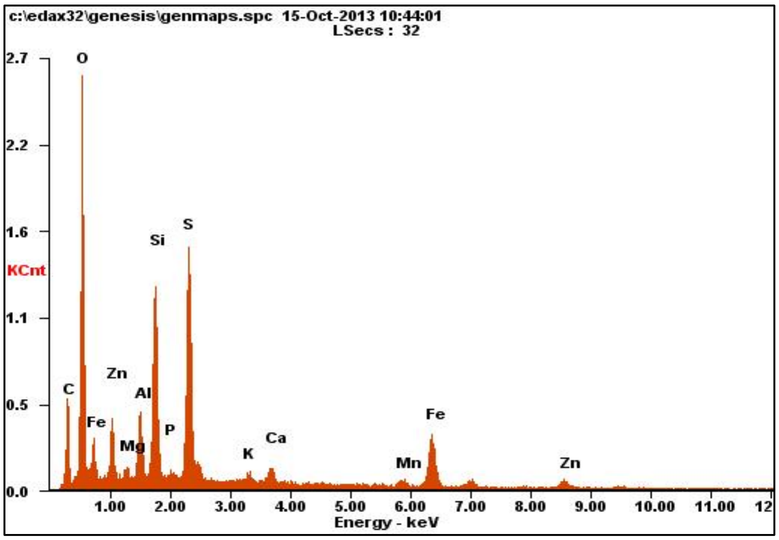
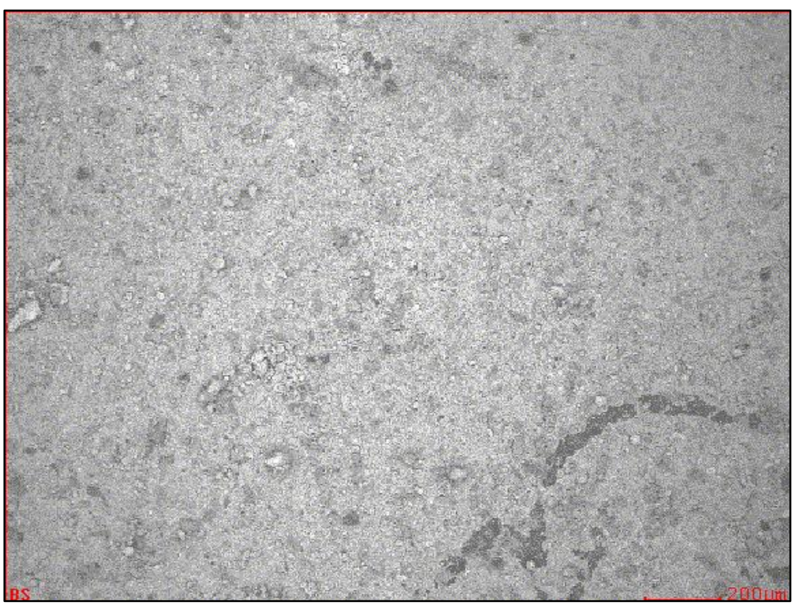


Element	***Wt%***	***At%***
CK	20.47	35.04
OK	30.87	39.68
MgK	00.52	00.44
AlK	02.62	02.00
SiK	08.26	06.05
PK	00.23	00.16
SK	11.36	07.29
KK	00.71	00.37
CaK	01.26	00.65
MnK	01.35	00.51
FeK	14.61	05.38
ZnK	07.72	02.43
Matrix	Correction	ZAF



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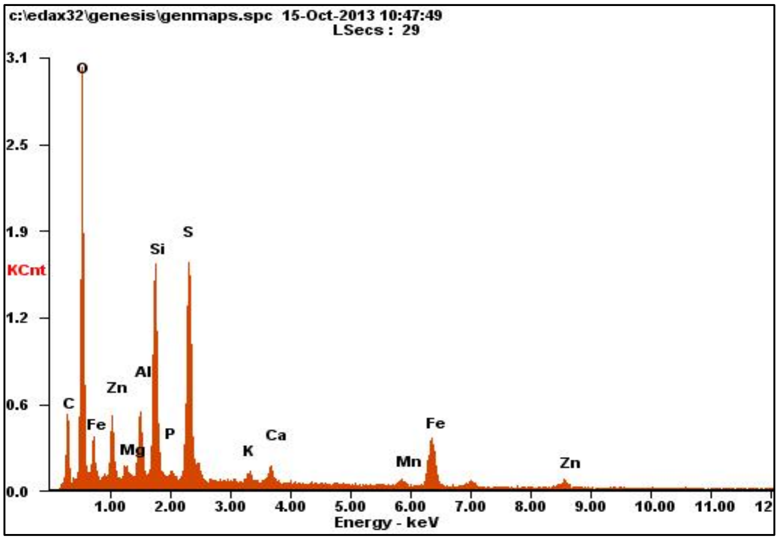
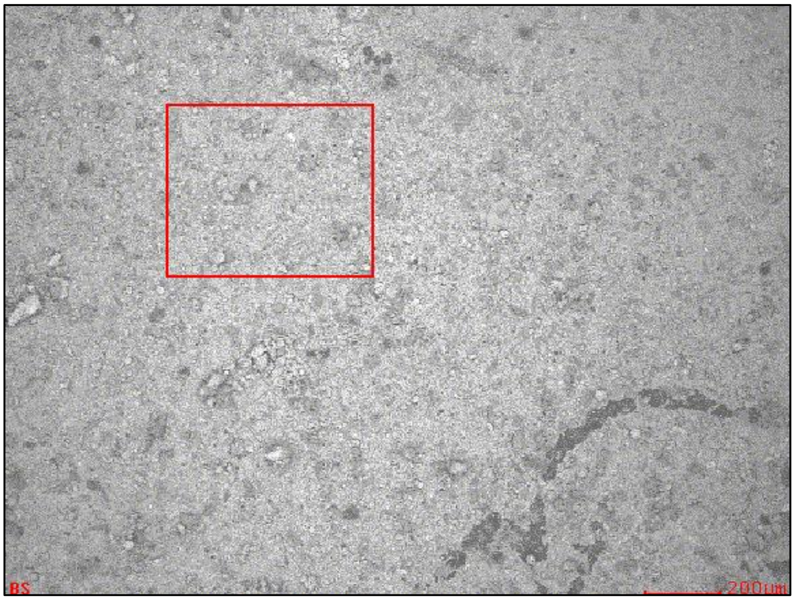


Element	***Wt%***	***At%***
CK	18.39	32.14
OK	31.17	40.91
MgK	00.59	00.51
AlK	02.92	02.27
SiK	09.07	06.78
PK	00.19	00.13
SK	11.44	07.49
KK	00.87	00.47
CaK	01.29	00.67
MnK	01.61	00.61
FeK	14.70	05.53
ZnK	07.77	02.50
Matrix	Correction	ZAF



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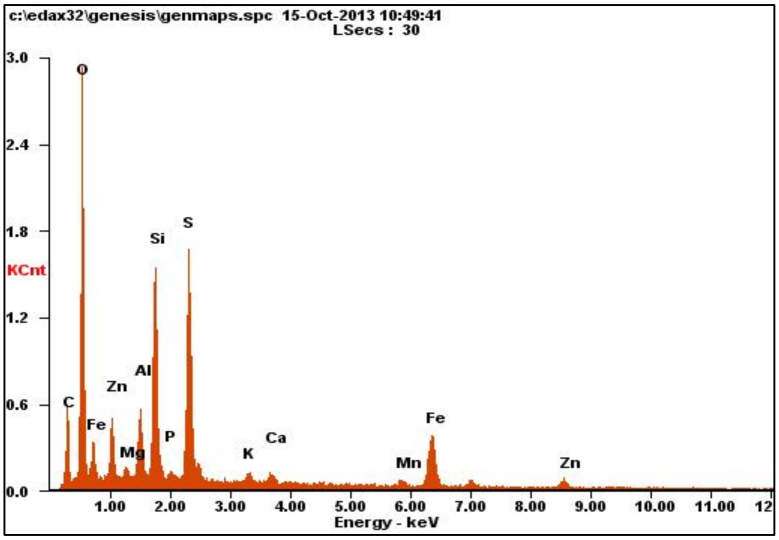
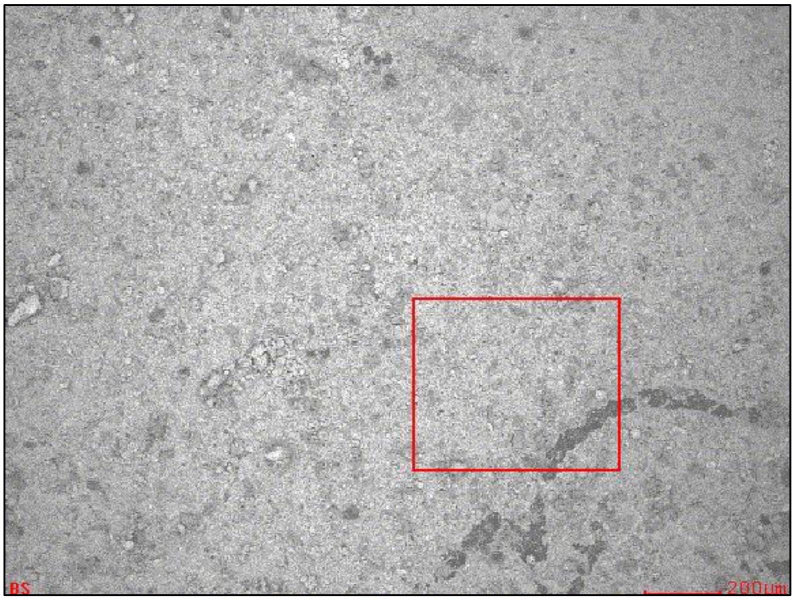


Element	*Wt%*	*At%*
CK	19.10	33.41
OK	30.38	39.90
MgK	00.48	00.41
AlK	02.76	02.15
SiK	09.09	06.80
PK	00.27	00.18
SK	11.00	07.20
KK	00.89	00.48
CaK	00.85	00.44
MnK	01.54	00.59
FeK	15.25	05.74
ZnK	08.41	02.70
Matrix	Correction	ZAF



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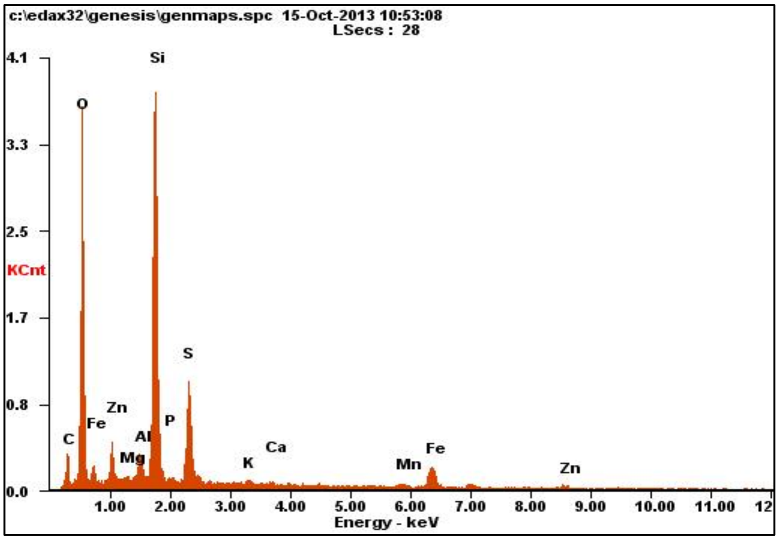
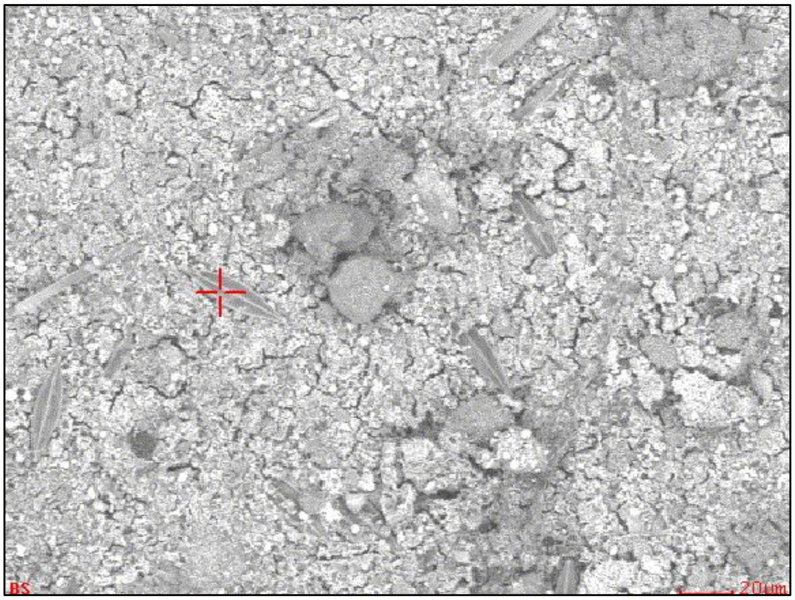


Element	*Wt%*	*At%*
CK	12.95	22.14
OK	37.51	48.15
MgK	00.29	00.24
AlK	01.53	01.17
SiK	23.63	17.28
PK	00.36	00.24
SK	07.86	05.04
KK	00.51	00.27
CaK	00.41	00.21
MnK	01.06	00.40
FeK	09.46	03.48
ZnK	04.43	01.39
Matrix	Correction	ZAF



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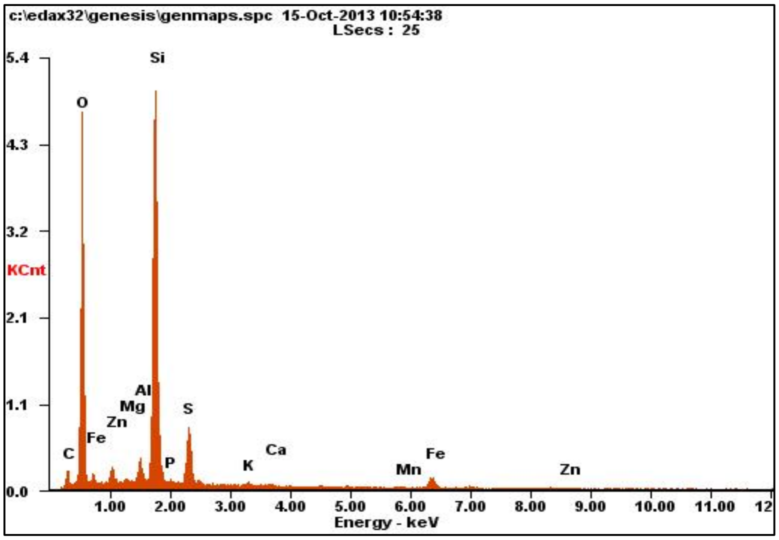
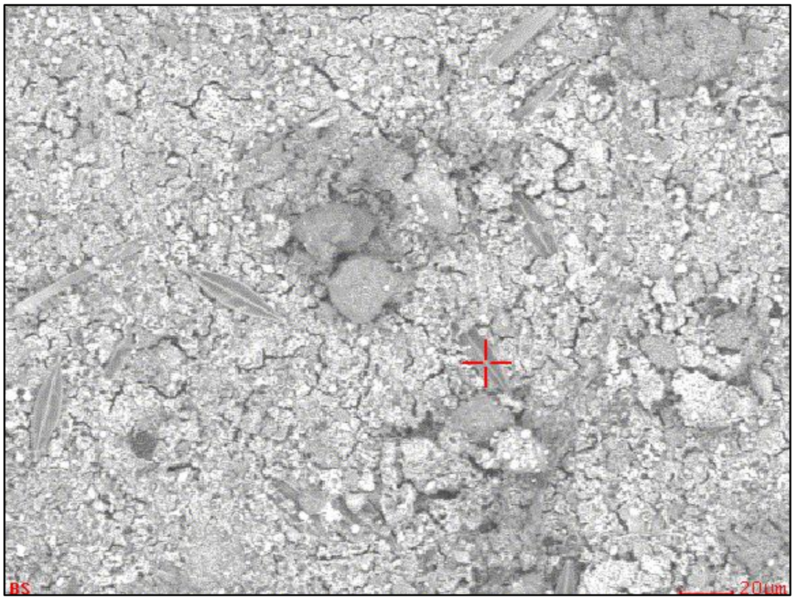


Element	*Wt%*	*At%*
CK	09.31	15.47
OK	44.60	55.63
MgK	00.15	00.12
AlK	01.47	01.09
SiK	28.93	20.56
PK	00.24	00.15
SK	05.72	03.56
KK	00.45	00.23
CaK	00.37	00.19
MnK	00.64	00.23
FeK	05.58	01.99
ZnK	02.54	00.78
Matrix	Correction	ZAF



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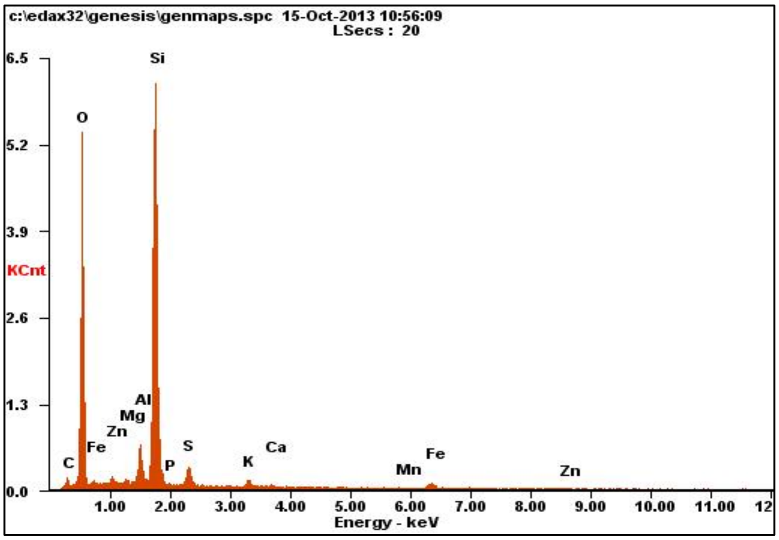
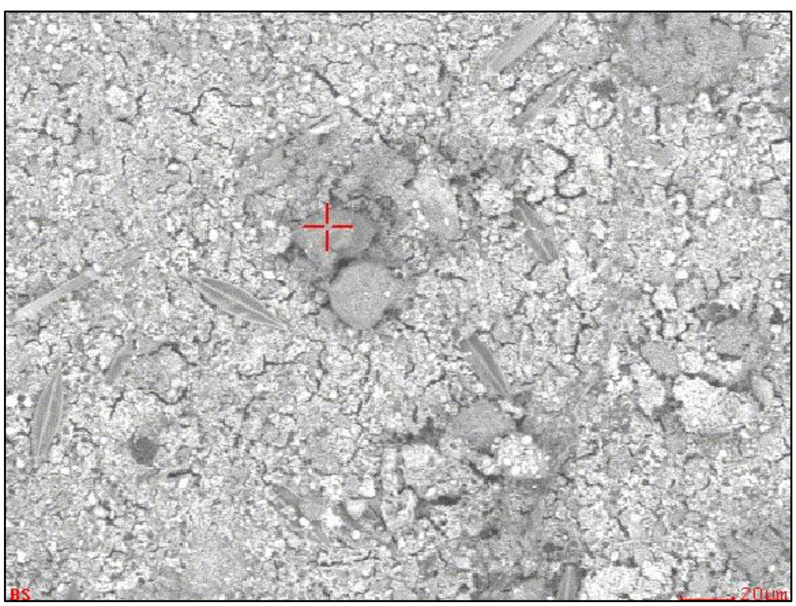


Element	*Wt%*	*At%*
CK	05.62	09.37
OK	47.52	59.50
MgK	00.27	00.22
AlK	02.92	02.16
SiK	34.49	24.60
PK	00.06	00.04
SK	02.49	01.56
KK	01.07	00.55
CaK	00.44	00.22
MnK	00.45	00.16
FeK	03.34	01.20
ZnK	01.32	00.41
Matrix	Correction	ZAF



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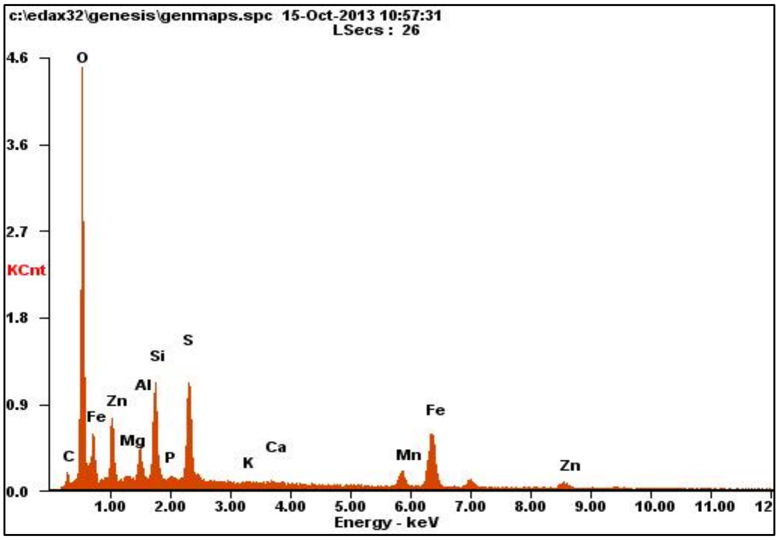
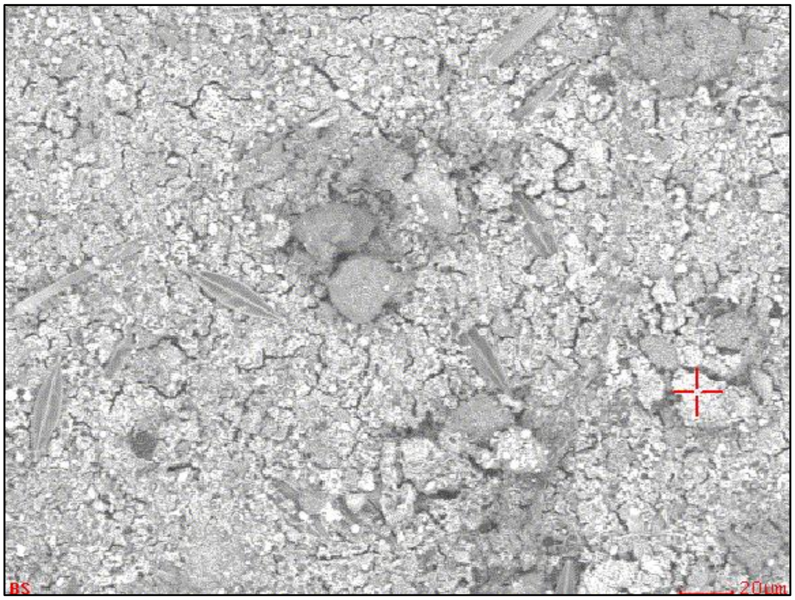


Element	*Wt%*	*At%*
CK	05.41	11.24
OK	35.53	55.45
MgK	00.43	00.44
AlK	02.39	02.21
SiK	07.05	06.26
PK	00.24	00.20
SK	08.29	06.45
KK	00.27	00.17
CaK	00.42	00.26
MnK	05.34	02.43
FeK	25.36	11.34
ZnK	09.26	03.54
Matrix	Correction	ZAF



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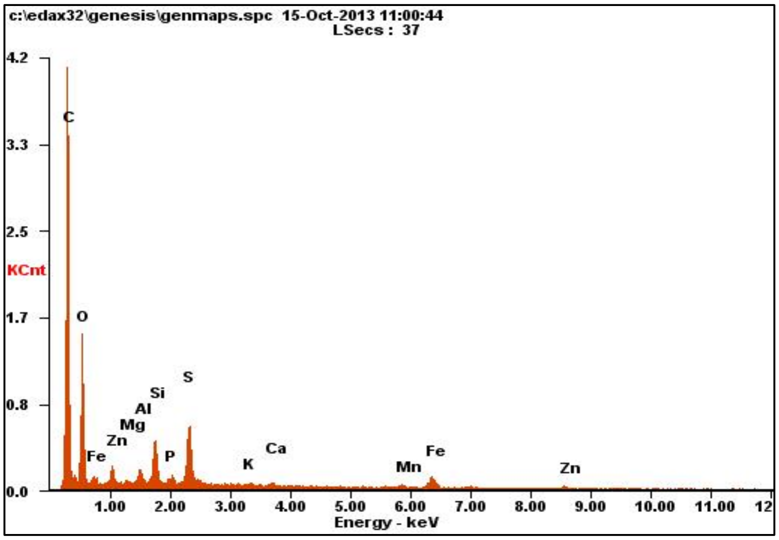
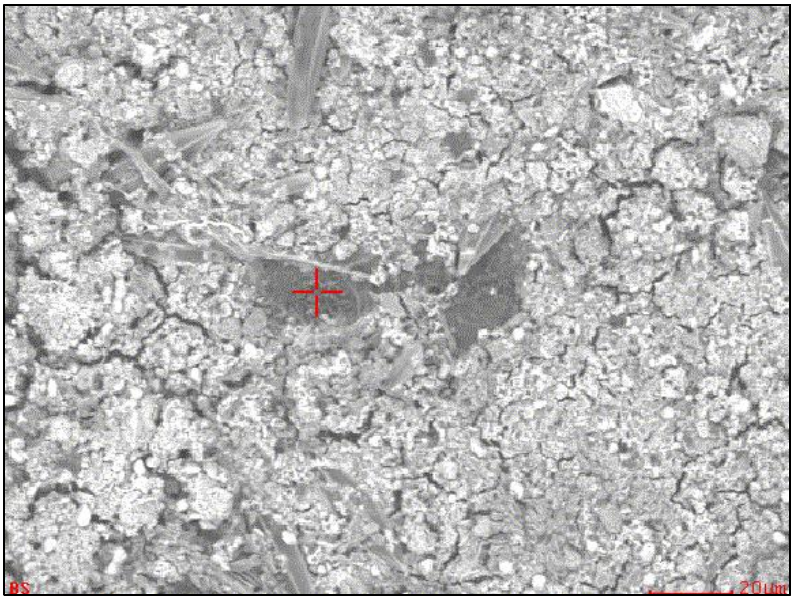


Element	Wt%	At%
CK	62.08	74.61
OK	21.82	19.69
MgK	00.16	00.10
AlK	00.69	00.37
SiK	02.23	01.15
PK	00.47	00.22
SK	03.52	01.58
KK	00.23	00.09
CaK	00.31	00.11
MnK	00.95	00.25
FeK	04.59	01.19
ZnK	02.93	00.65
Matrix	Correction	ZAF



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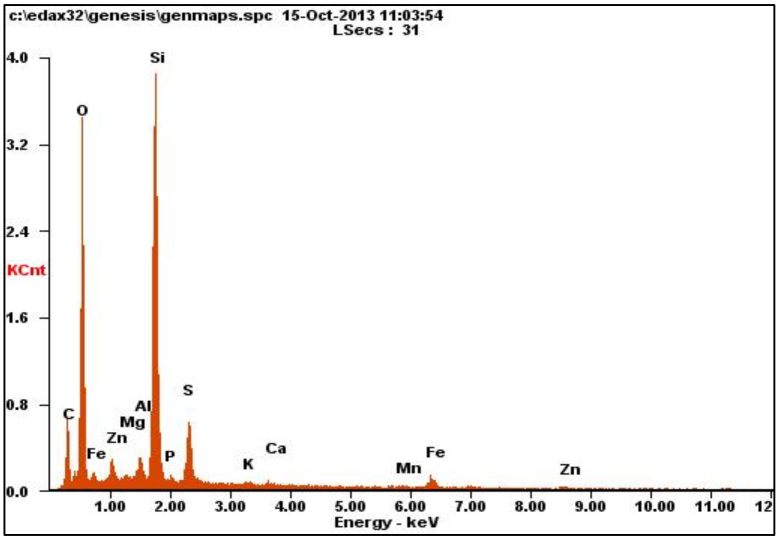
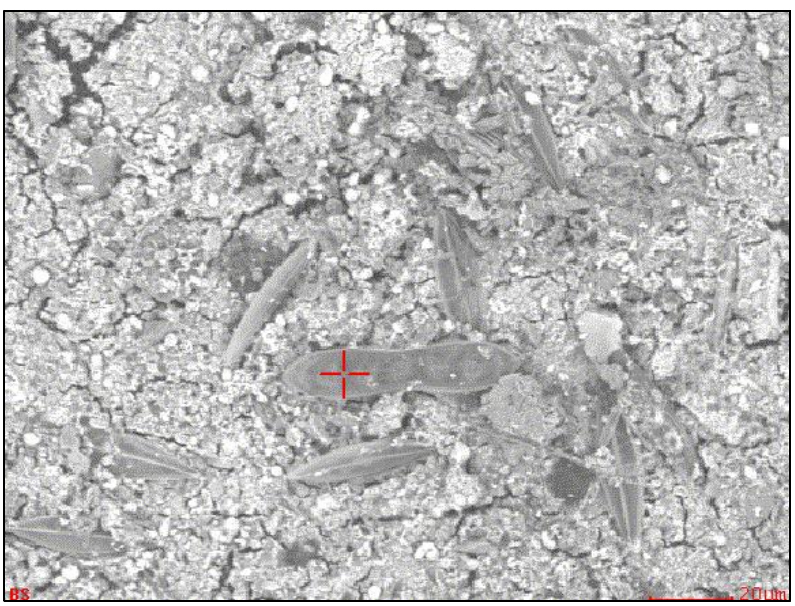


Element	*Wt%*	*At%*
CK	22.72	34.37
OK	38.89	44.17
MgK	00.29	00.22
AlK	01.20	00.81
SiK	22.72	14.70
PK	00.31	00.18
SK	04.54	02.57
KK	00.29	00.13
CaK	00.36	00.17
MnK	00.87	00.29
FeK	04.71	01.53
ZnK	03.09	00.86
Matrix	Correction	ZAF



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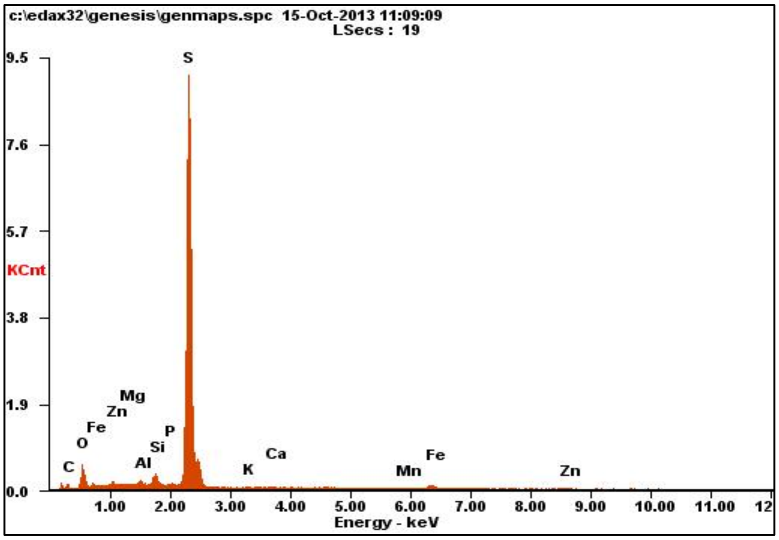
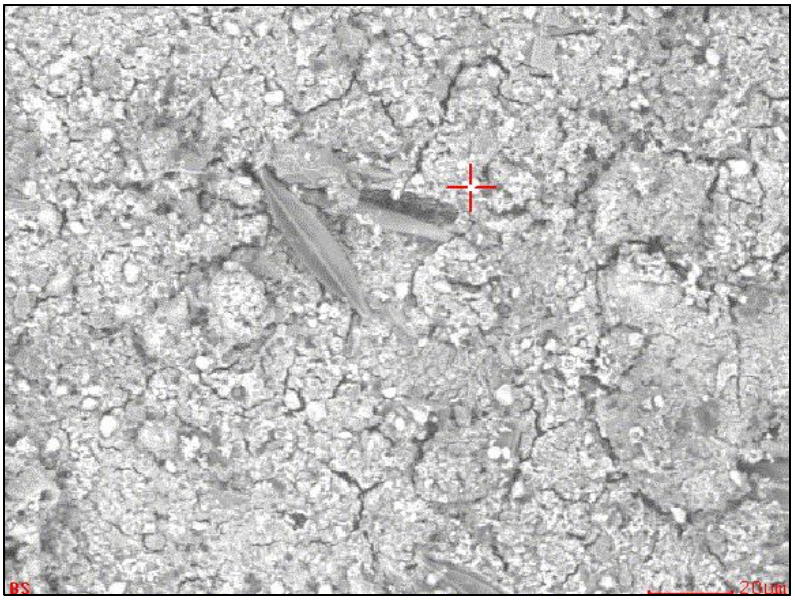


Element	*Wt%*	*At%*
CK	08.72	18.85
OK	11.45	18.58
MgK	00.27	00.28
AlK	00.57	00.55
SiK	01.63	01.51
PK	00.23	00.19
SK	70.19	56.83
KK	00.24	00.16
CaK	00.36	00.23
MnK	00.40	00.19
FeK	03.81	01.77
ZnK	02.14	00.85
Matrix	Correction	ZAF



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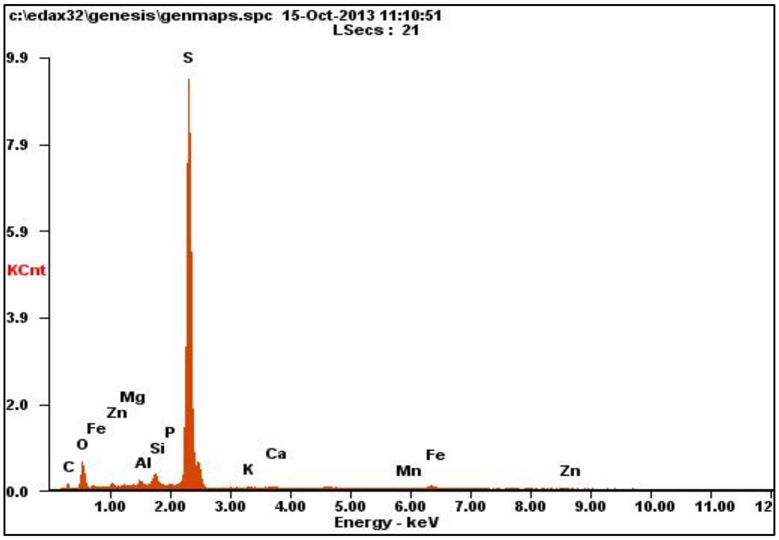
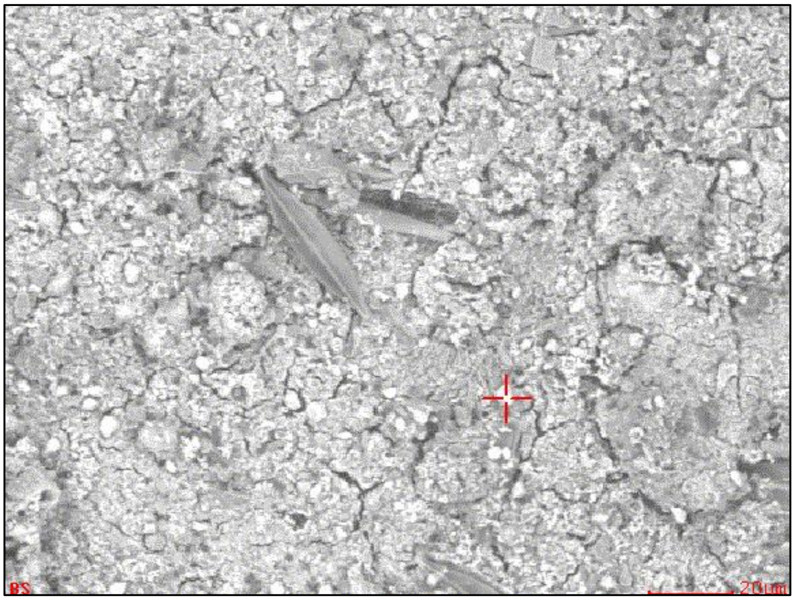


Element	*Wt%*	*At%*
CK	10.08	21.41
OK	11.82	18.84
MgK	00.10	00.11
AlK	00.53	00.50
SiK	01.51	01.37
PK	00.29	00.24
SK	67.71	53.86
KK	00.40	00.26
CaK	00.63	00.40
MnK	00.71	00.33
FeK	03.94	01.80
ZnK	02.28	00.89
Matrix	Correction	ZAF



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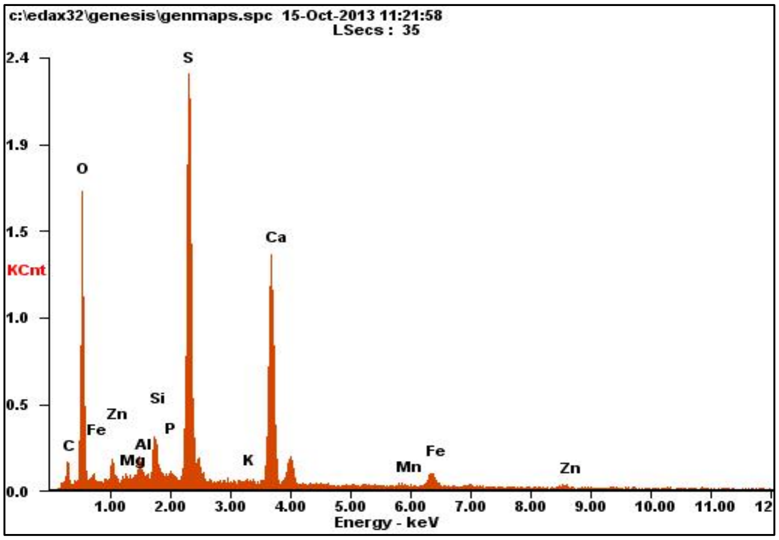
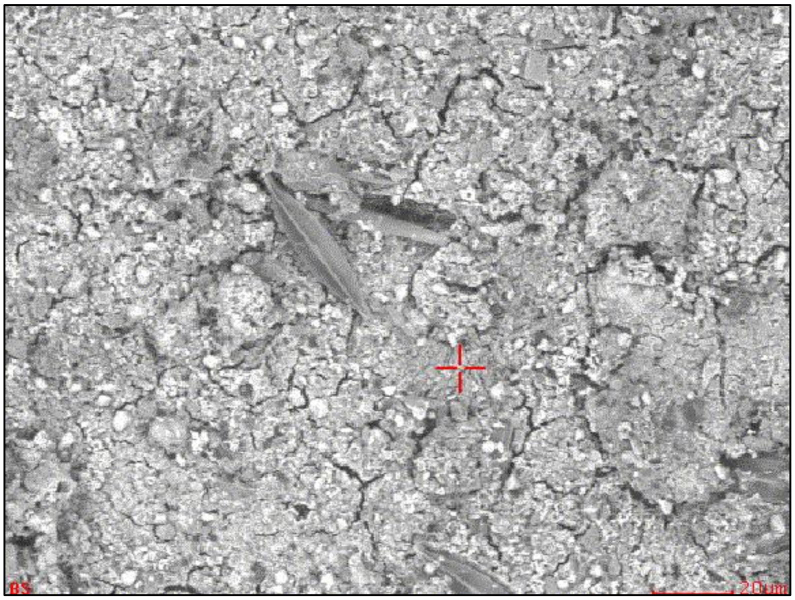


Element	Wt%	At%
CK	07.64	14.38
OK	36.87	52.11
MgK	00.09	00.08
AlK	00.76	00.64
SiK	01.99	01.60
PK	00.20	00.14
SK	21.80	15.38
KK	00.34	00.20
CaK	21.25	11.99
MnK	00.73	00.30
FeK	04.89	01.98
ZnK	03.45	01.19
Matrix	Correction	ZAF



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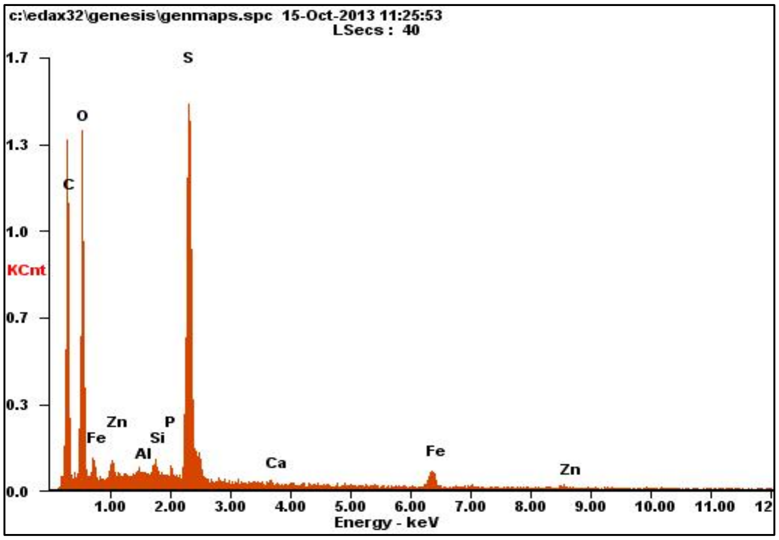
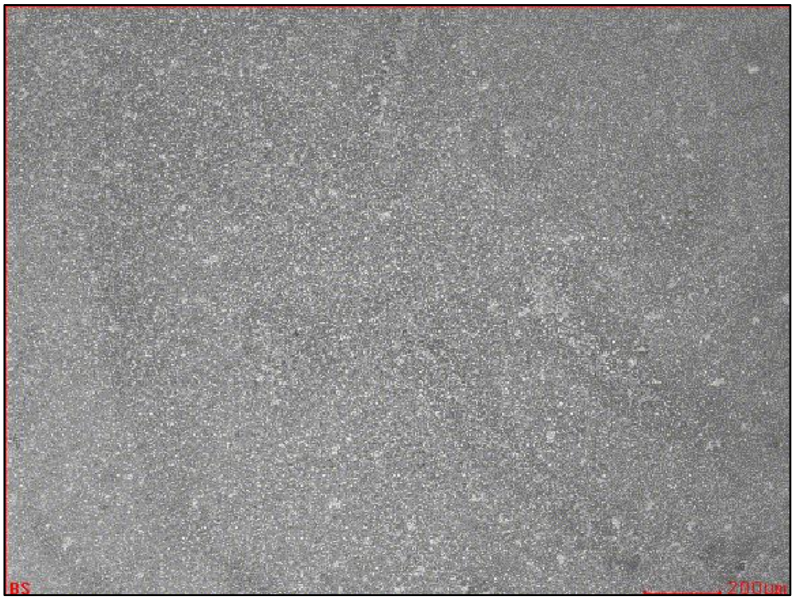


Element	*Wt%*	*At%*
CK	49.10	63.05
OK	28.66	27.63
AlK	00.22	00.12
SiK	00.52	00.29
PK	00.26	00.13
SK	14.48	06.97
CaK	00.29	00.11
FeK	04.36	01.20
ZnK	02.12	00.50
Matrix	Correction	ZAF



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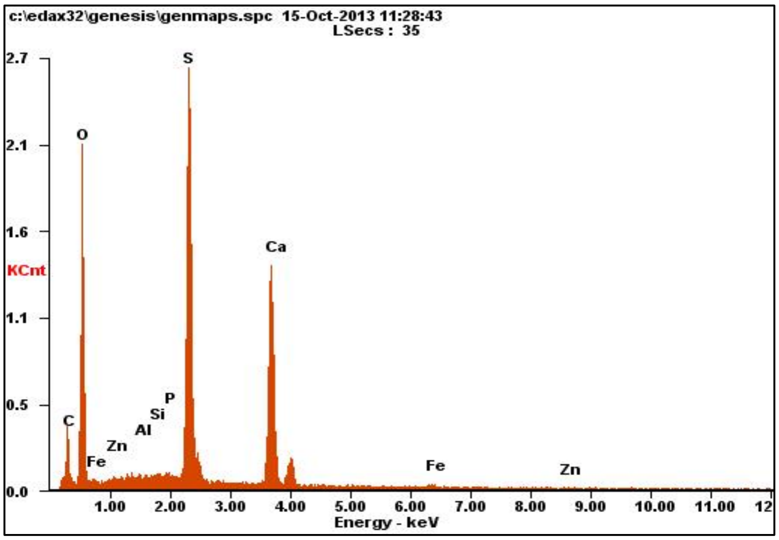
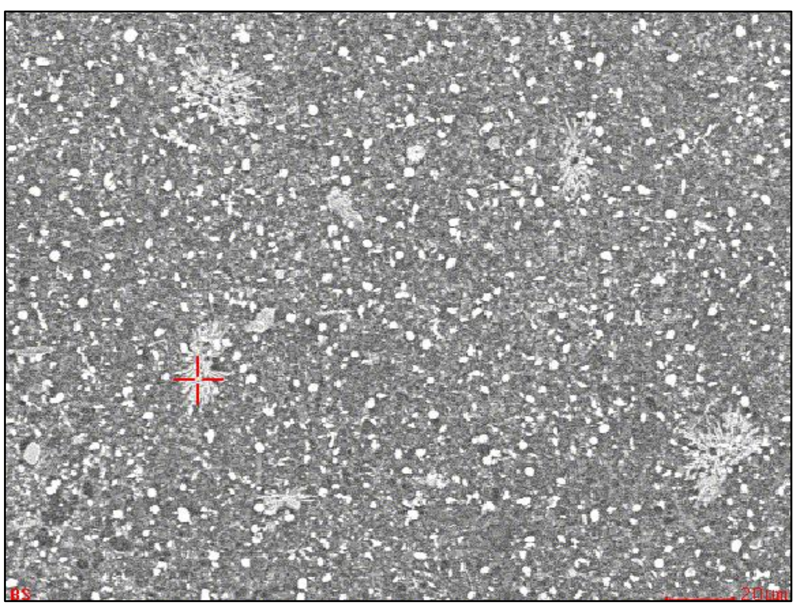


Element	*Wt%*	*At%*
CK	15.25	25.07
OK	41.92	51.75
AlK	00.00	00.00
SiK	00.09	00.06
PK	00.07	00.05
SK	20.25	12.47
CaK	19.76	09.74
FeK	01.16	00.41
ZnK	01.49	00.45
Matrix	Correction	ZAF



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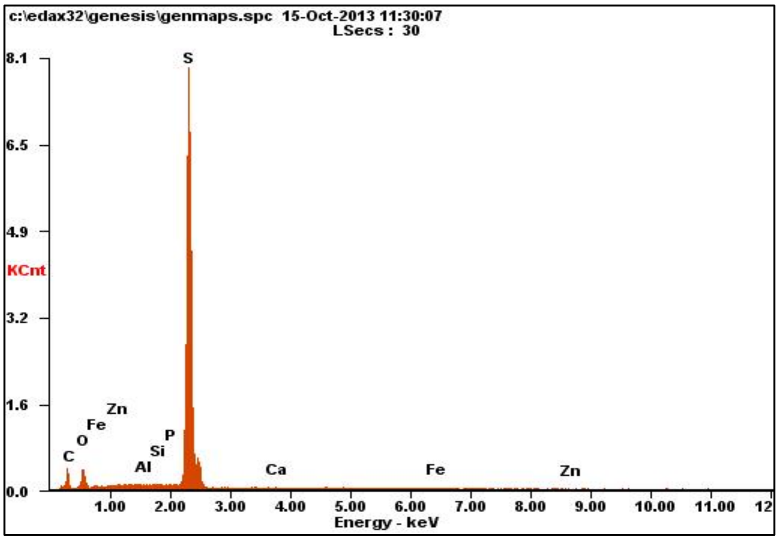
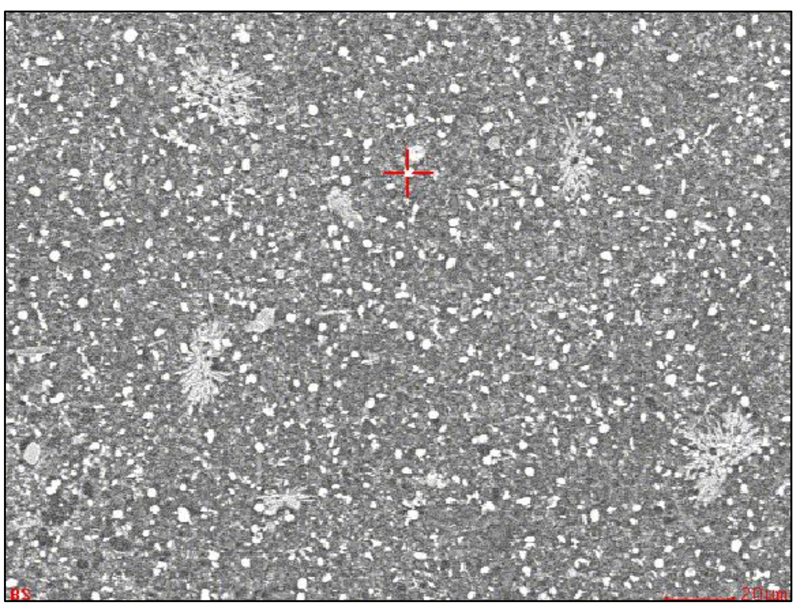


Element	Wt%	At%
CK	29.11	49.57
OK	08.91	11.39
AlK	00.10	00.07
SiK	00.20	00.15
PK	00.28	00.19
SK	59.49	37.95
CaK	00.19	00.10
FeK	01.02	00.37
ZnK	00.69	00.22
Matrix	Correction	ZAF



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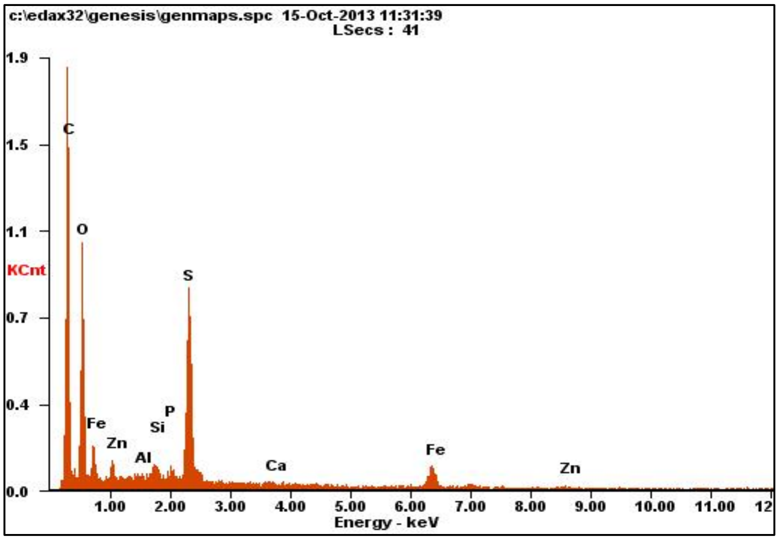
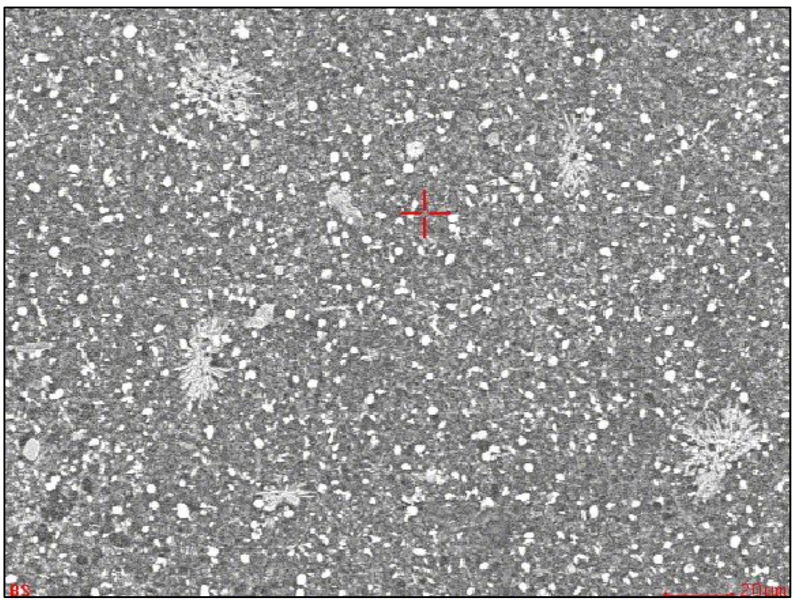


Element	*Wt%*	*At%*
CK	56.20	69.99
OK	24.52	22.92
AlK	00.17	00.09
SiK	00.58	00.31
PK	00.44	00.21
SK	08.52	03.97
CaK	00.32	00.12
FeK	06.73	01.80
ZnK	02.52	00.58
Matrix	Correction	ZAF



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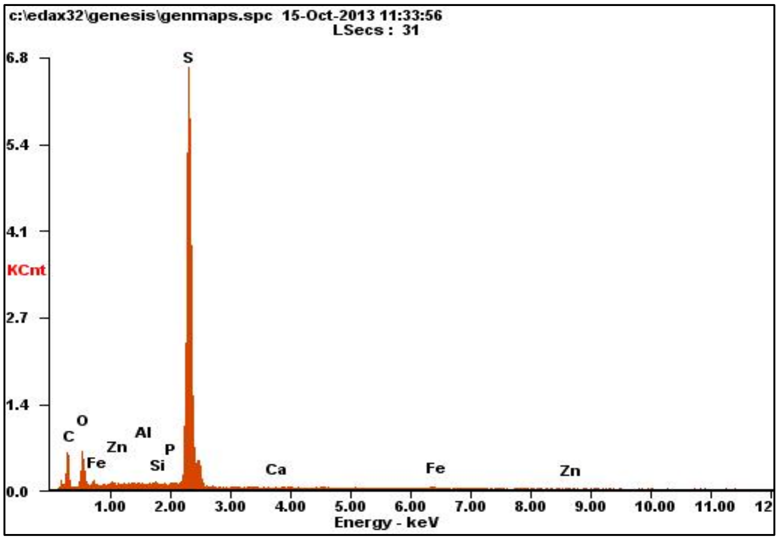
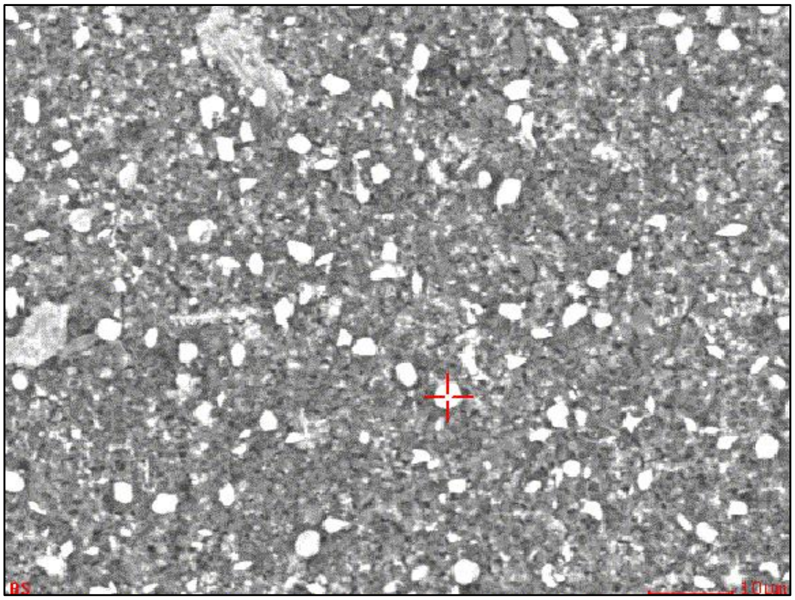


Element	***Wt%***	***At%***
CK	37.38	57.60
OK	11.90	13.77
AlK	00.08	00.06
SiK	00.27	00.18
PK	00.29	00.17
SK	47.35	27.34
CaK	00.13	00.06
FeK	01.78	00.59
ZnK	00.82	00.23
Matrix	Correction	ZAF



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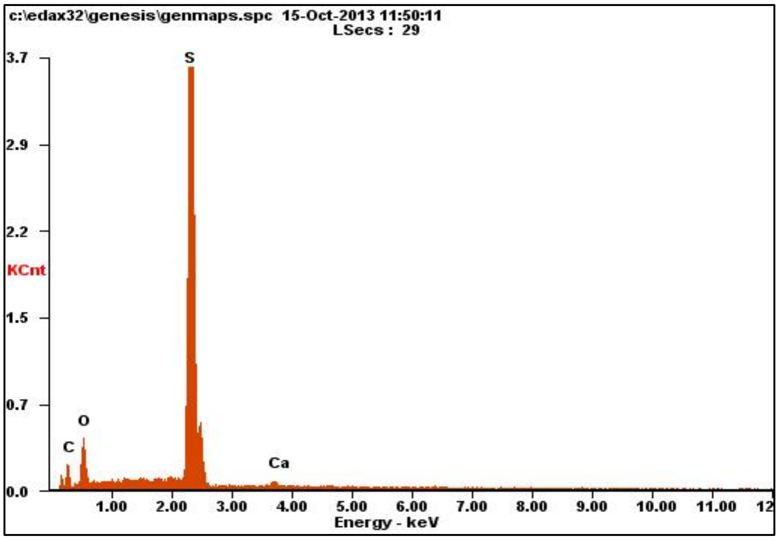

Element	*Wt%*	*At%*
CK	22.66	40.69
OK	11.01	14.84
SK	65.38	43.97
CaK	00.94	00.50
Matrix	Correction	ZAF

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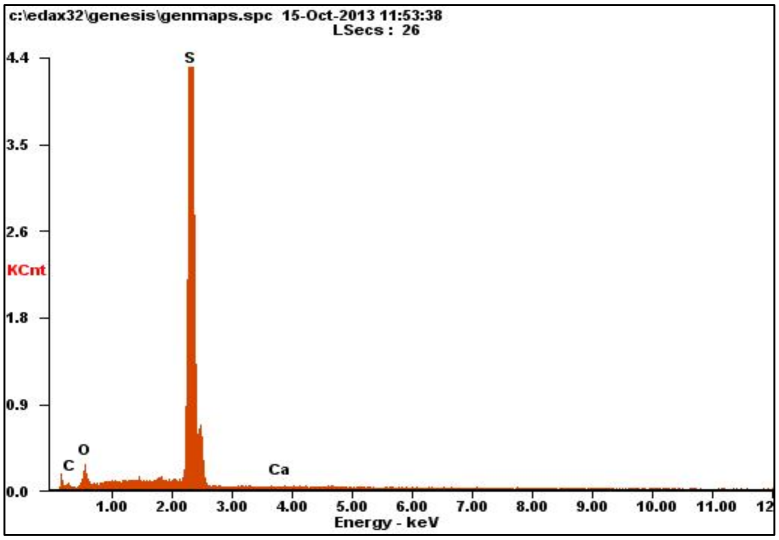
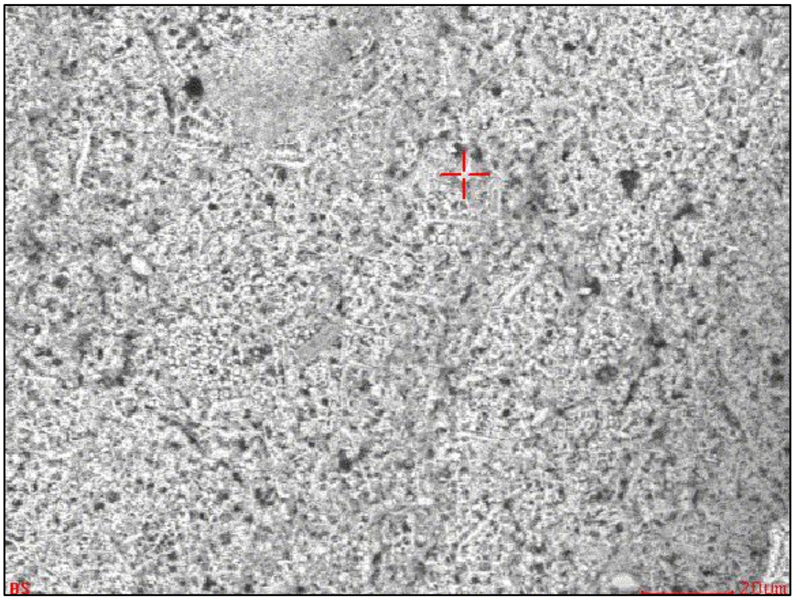


Element	*Wt%*	*At%*
CK	07.00	16.14
OK	04.10	07.10
SK	88.71	76.63
CaK	00.19	00.13
Matrix	Correction	ZAF



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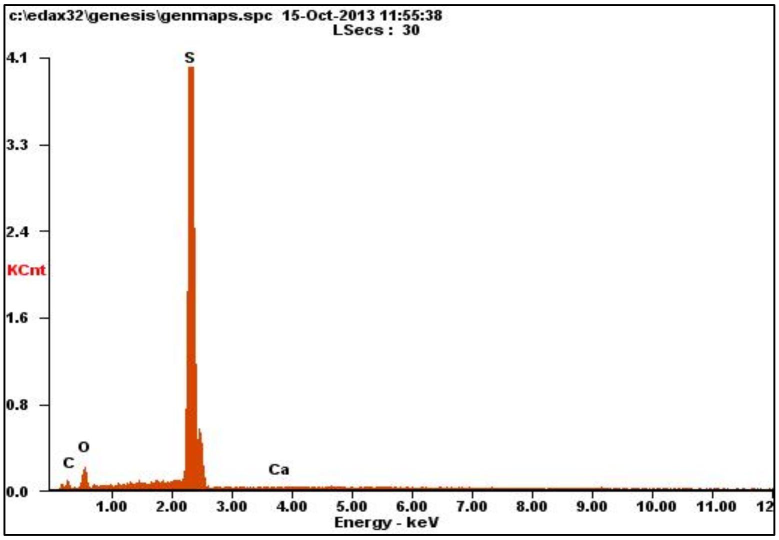
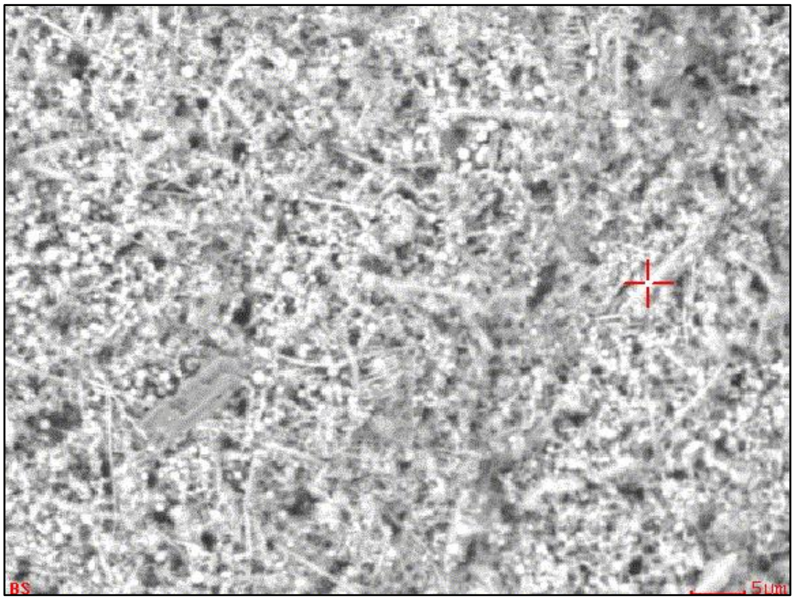


Element	*Wt%*	*At%*
CK	11.14	23.96
OK	05.49	08.87
SK	83.14	67.01
CaK	00.24	00.15
Matrix	Correction	ZAF



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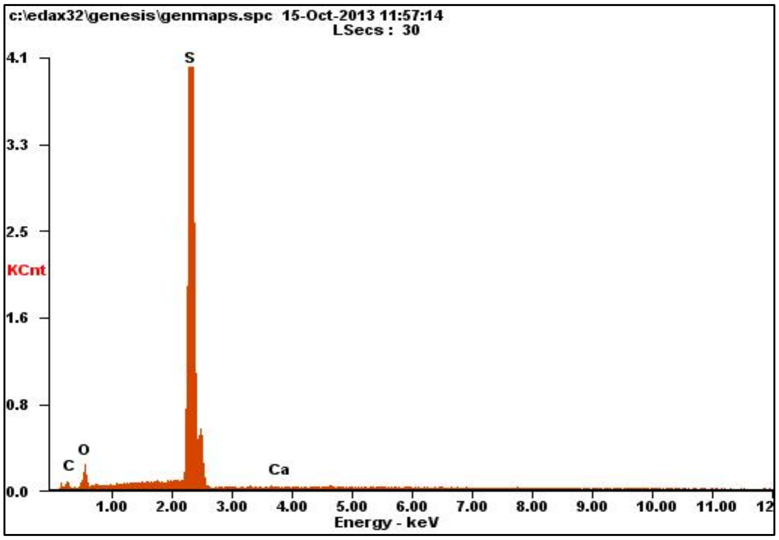
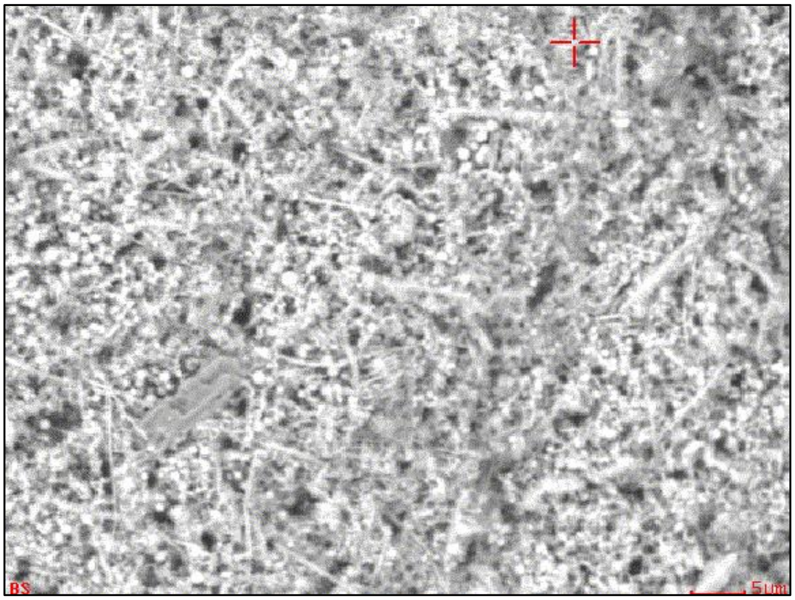


Element	*Wt%*	*At%*
CK	09.88	21.81
OK	04.46	07.39
SK	85.35	70.59
CaK	00.31	00.21
Matrix	Correction	ZAF



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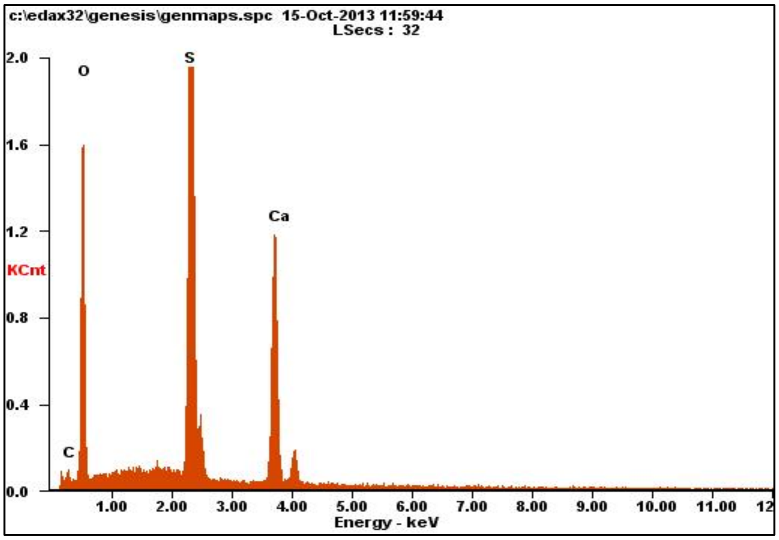
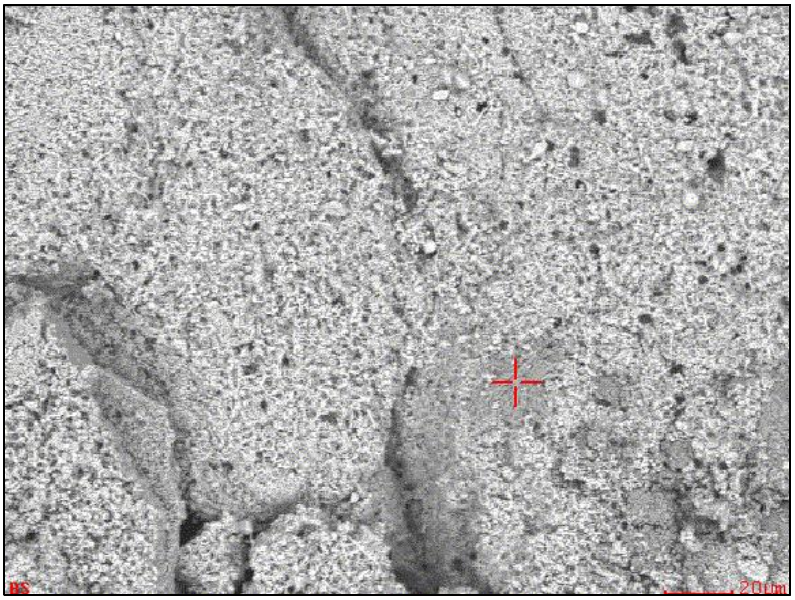


Element	***Wt%***	***At%***
CK	04.58	08.49
OK	40.19	55.91
SK	35.53	24.67
CaK	19.69	10.94
Matrix	Correction	ZAF



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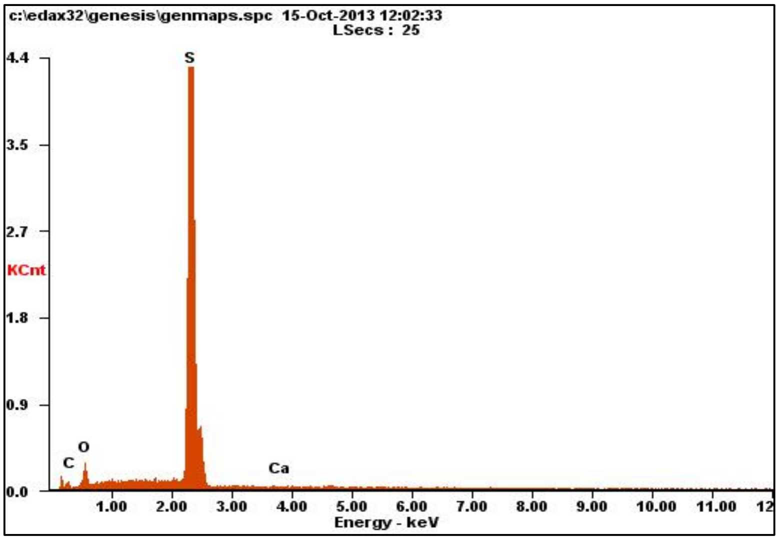
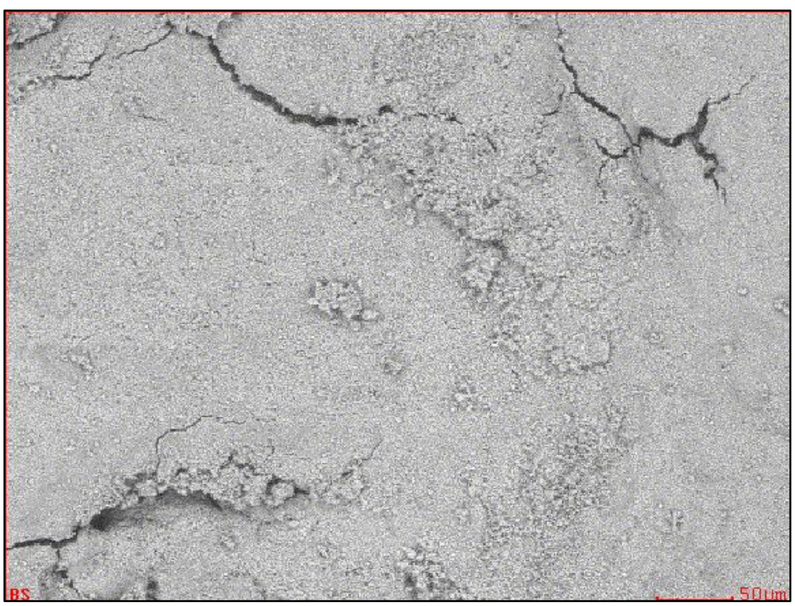


Element	*Wt%*	*At%*
CK	08.58	19.35
OK	04.05	06.86
SK	87.00	73.54
CaK	00.37	00.25
Matrix	Correction	ZAF



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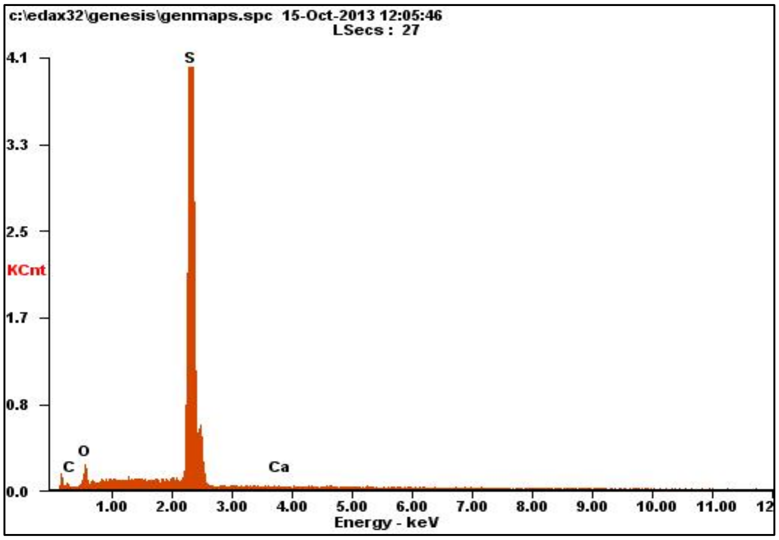
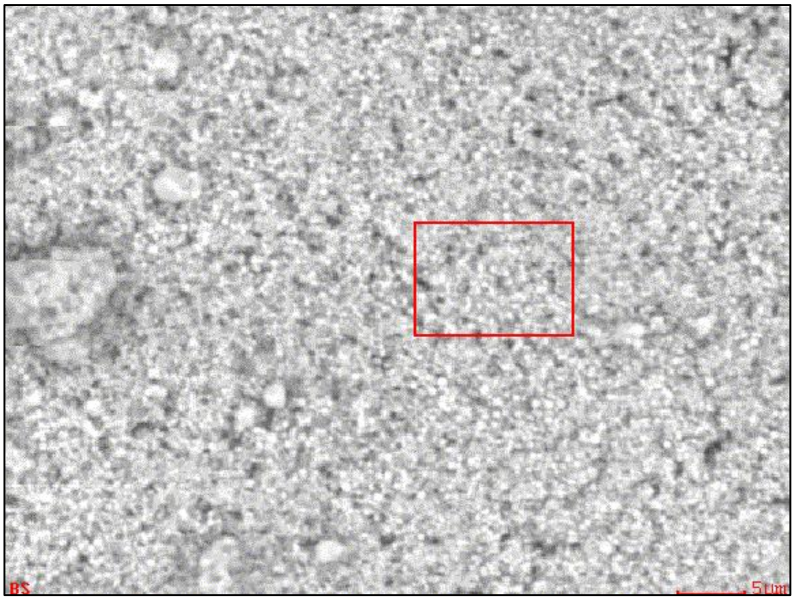


Element	*Wt%*	*At%*
CK	05.59	13.29
OK	03.01	05.37
SK	91.15	81.16
CaK	00.25	00.18
Matrix	Correction	ZAF



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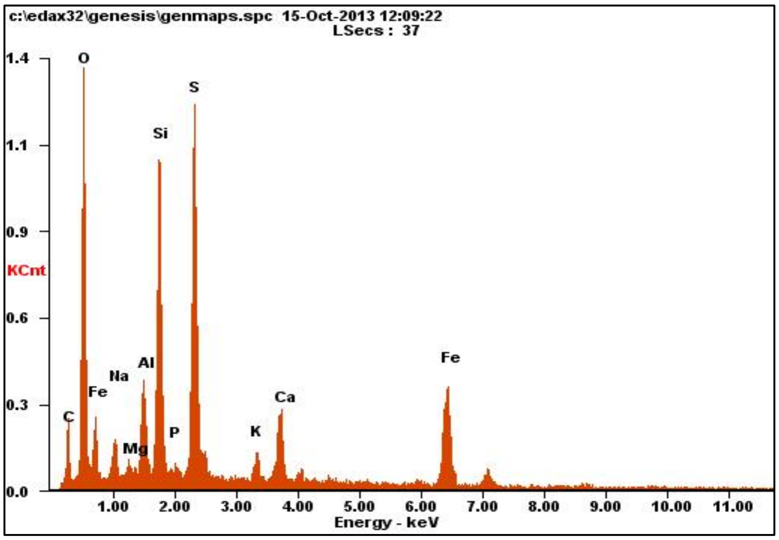
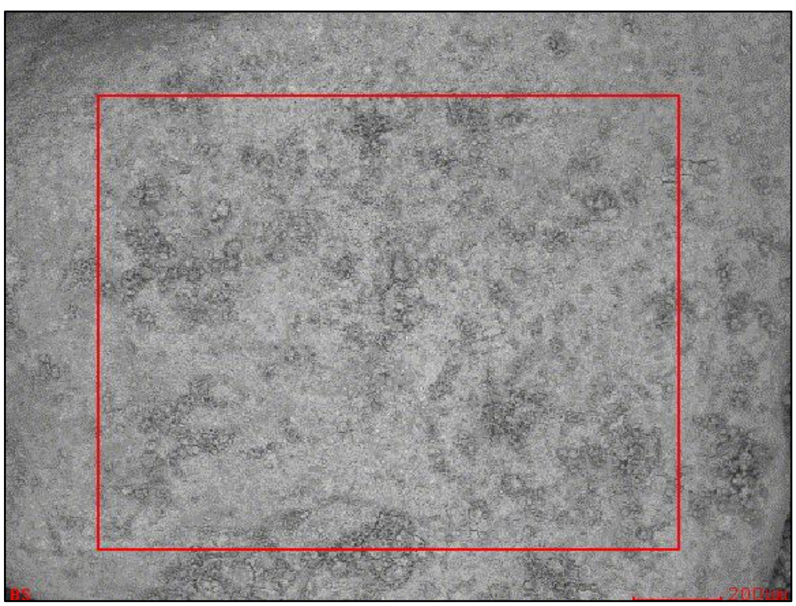


Element	*Wt%*	*At%*
CK	13.44	25.32
OK	27.58	39.01
NaK	01.63	01.61
MgK	00.44	00.41
AlK	03.09	02.59
SiK	10.12	08.15
PK	00.26	00.19
SK	13.85	09.77
KK	01.45	00.84
CaK	04.41	02.49
FeK	23.73	09.62
Matrix	Correction	ZAF



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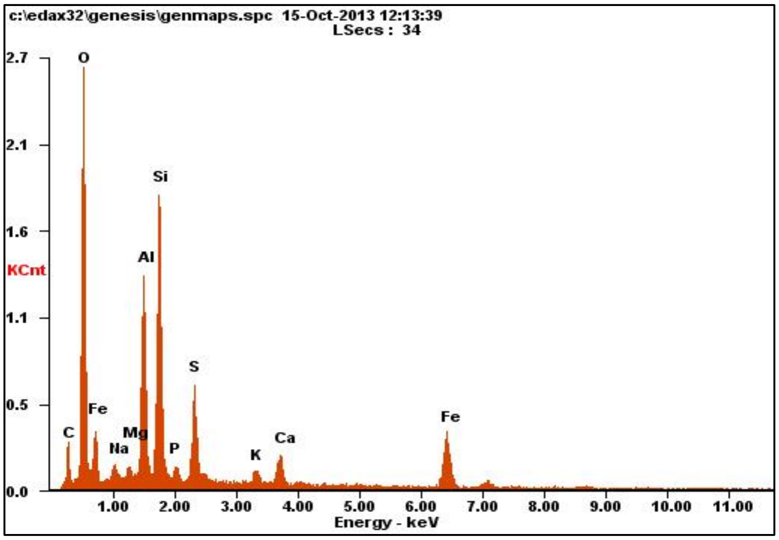
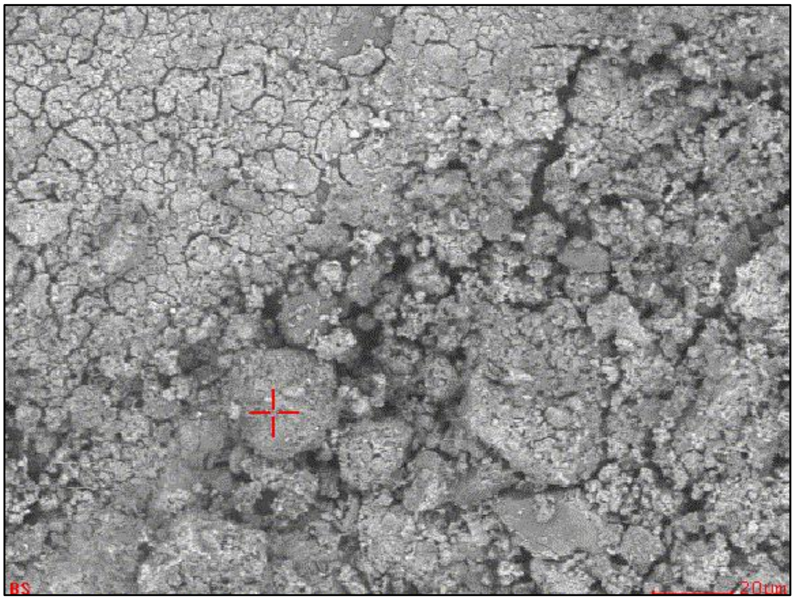


Element	*Wt%*	*At%*
CK	12.45	21.64
OK	36.45	47.58
NaK	00.81	00.74
MgK	00.58	00.50
AlK	09.08	07.03
SiK	13.91	10.34
PK	00.63	00.43
SK	05.25	03.42
KK	01.08	00.58
CaK	02.39	01.25
FeK	17.35	06.49
Matrix	Correction	ZAF



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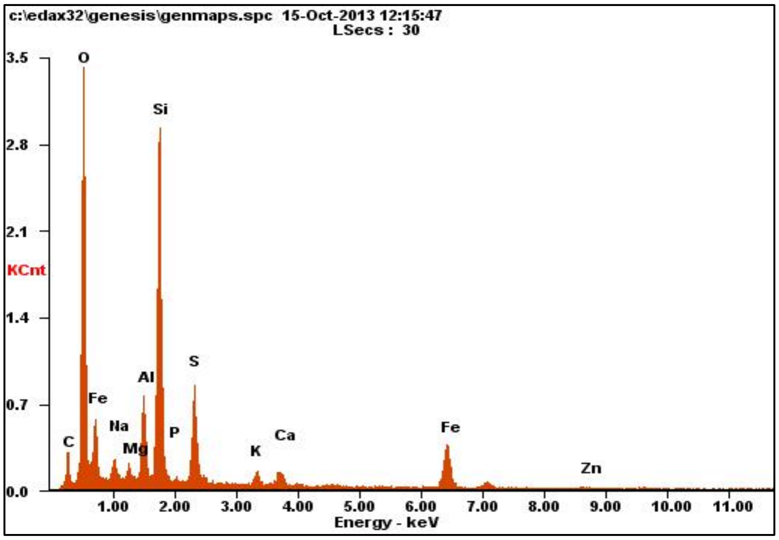
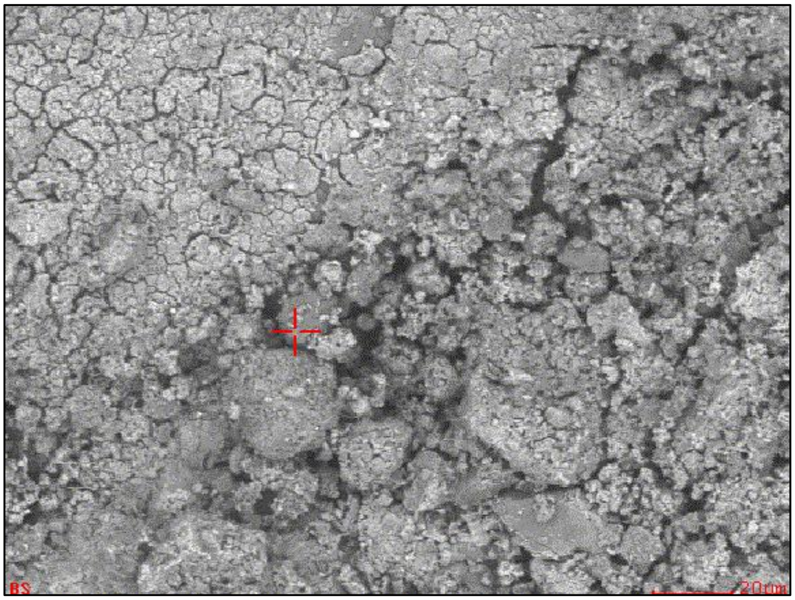


Element	*Wt%*	*At%*
CK	11.17	19.56
OK	38.07	50.07
NaK	01.14	01.05
MgK	00.69	00.59
AlK	04.03	03.14
SiK	17.83	13.36
PK	00.12	00.08
SK	05.98	03.93
KK	01.27	00.69
CaK	01.67	00.88
FeK	15.46	05.83
ZnK	02.57	00.83
Matrix	Correction	ZAF



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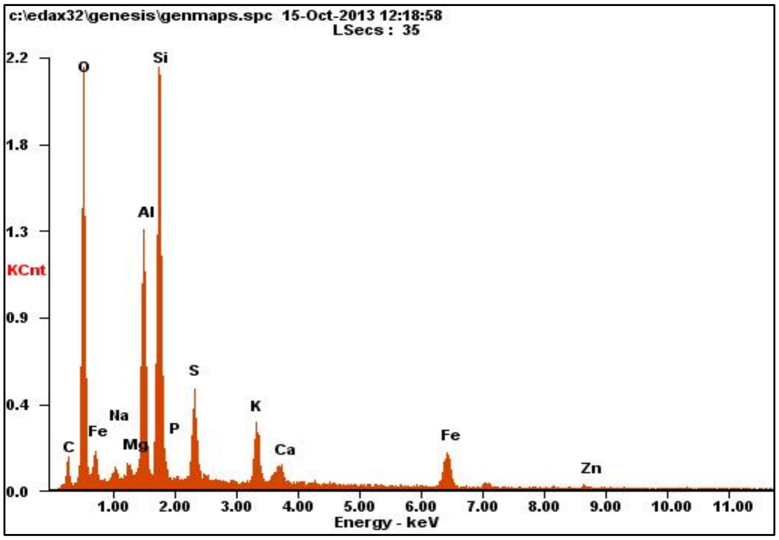
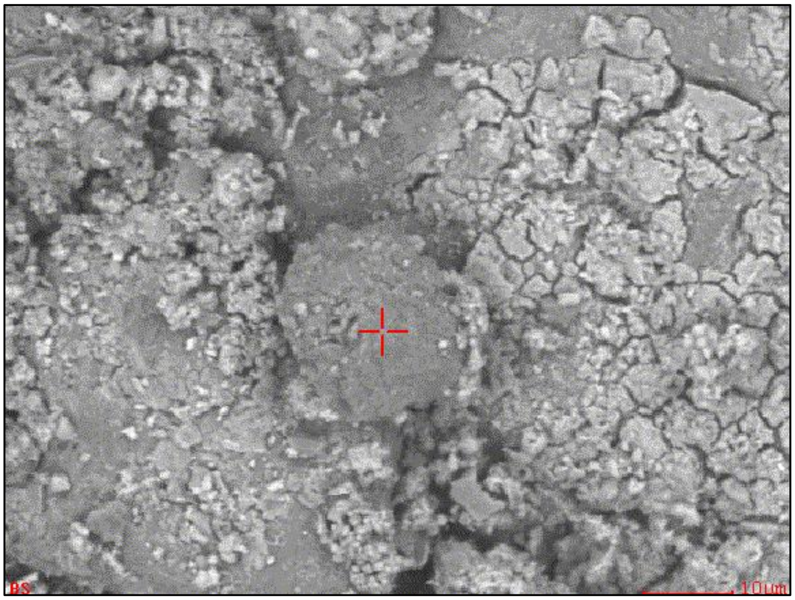


Element	*Wt%*	*At%*
CK	08.51	15.55
OK	33.66	46.20
NaK	00.65	00.63
MgK	00.81	00.73
AlK	10.69	08.70
SiK	20.17	15.77
PK	00.14	00.10
SK	04.99	03.42
KK	04.62	02.59
CaK	01.67	00.92
FeK	11.41	04.49
ZnK	02.68	00.90
Matrix	Correction	ZAF



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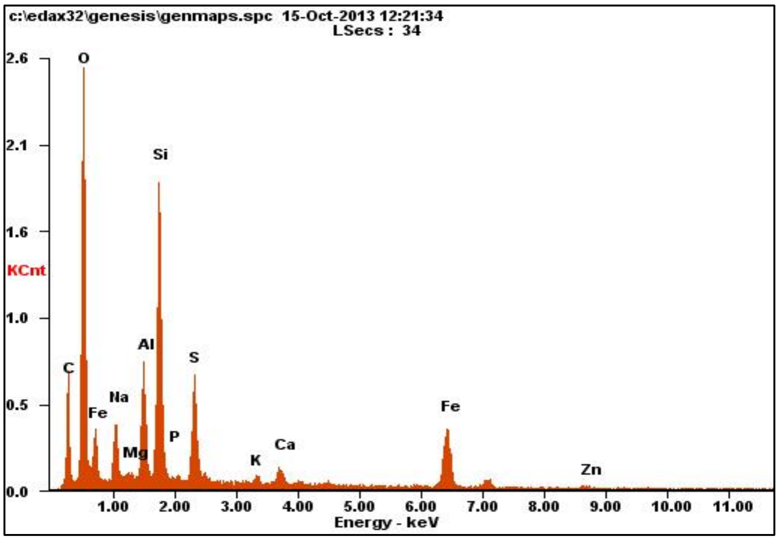
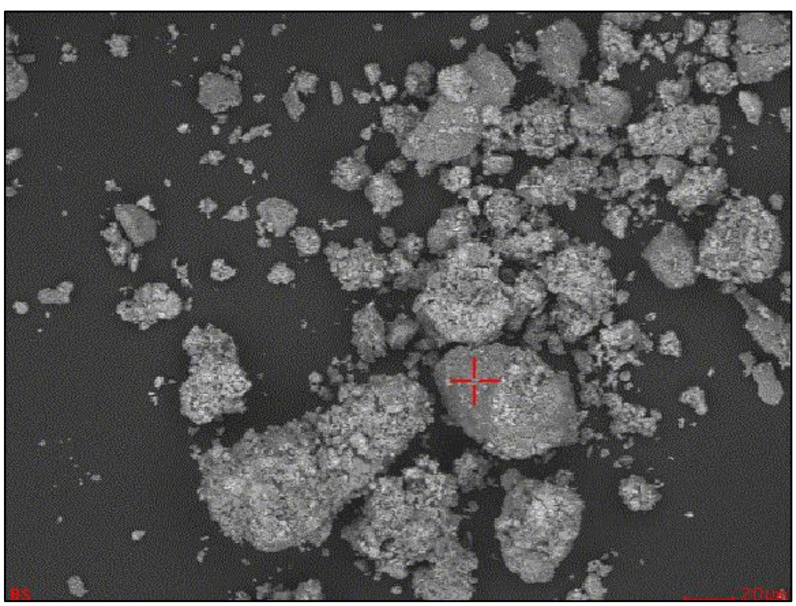


Element	*Wt%*	*At%*
CK	22.61	36.61
OK	31.94	38.82
NaK	03.06	02.59
MgK	00.20	00.16
AlK	04.37	03.15
SiK	11.79	08.16
PK	00.10	00.06
SK	04.91	02.98
KK	00.53	00.26
CaK	01.44	00.70
FeK	16.49	05.74
ZnK	02.55	00.76
Matrix	Correction	ZAF



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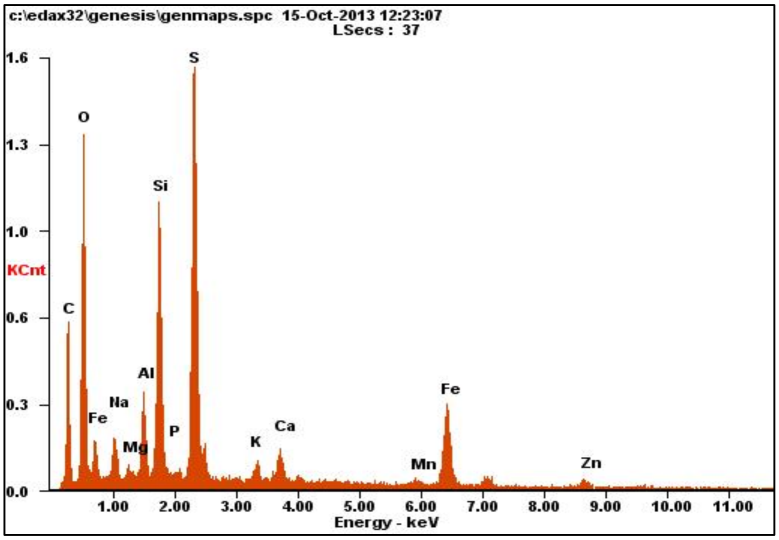
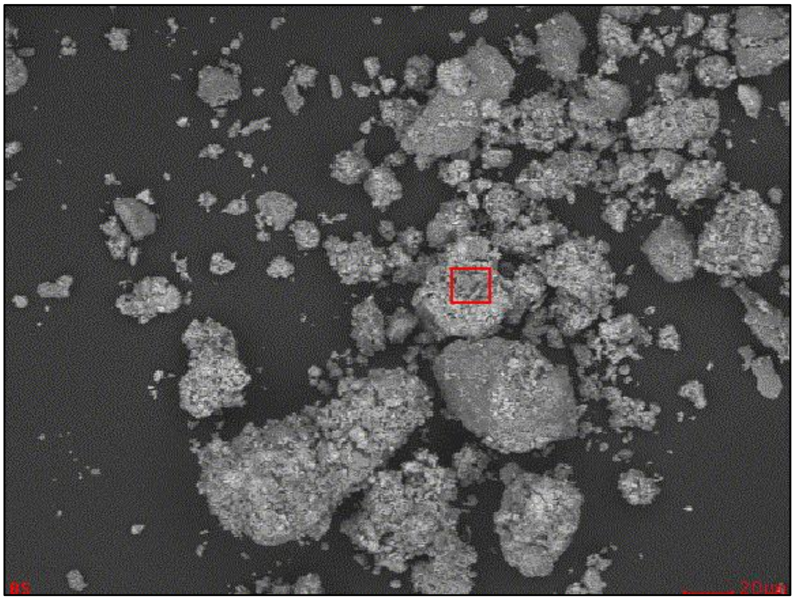


Element	*Wt%*	*At%*
CK	29.08	47.97
OK	21.16	26.21
NaK	01.41	01.21
MgK	00.35	00.29
AlK	02.08	01.53
SiK	07.41	05.23
PK	00.16	00.10
SK	14.31	08.84
KK	00.90	00.45
CaK	01.60	00.79
MnK	00.72	00.26
FeK	15.54	05.51
ZnK	05.27	01.60
Matrix	Correction	ZAF



Atlantic Richfield Company
A BP affiliated company

Laboratory Management Program LaMP Chain of Custody Record

BP/ARC Project Name: Rico-Argentine Mine Site
BP/ARC Facility No:
Req Due Date (mm/dd/yy):
Rush TAT: Yes ___ No X
Lab Work Order Number:

Lab Name:	Southern Methodist University, Dept. of Earth Sciences Lab
Lab Address:	3225 Daniel Avenue, Dallas, TX 75205
Lab PM:	Roy Beavers, Lab Manager
Lab Phone:	(214) 768-2756
Lab Shipping Accnt:	NA
Lab Bottle Order No:	NA
Other Info:	Wetland Pilot Scale Study
BP/ARC EBM:	Anthony Brown
EBM Phone:	714-228-6770
EBM Email:	anthony.brown@bp.com

BP/ARC Facility Address:	Rico-Argentine Mine
City, State, ZIP Code:	Rico, Colorado 81332
Lead Regulatory Agency:	U.S. EPA Region 8
California Global ID No.:	NA
Enfos Proposal No:	D009J-0066 (WR 268174)
Accounting Mode:	Provision X OOC-BU ___ OOC-RM ___
Stage: 4-Execute	Activity: Spend

Consultant/Contractor:	AMEC E&I, Inc.
Consultant/Contractor Project No:	SA11161314.400B
Address:	10670 White Rock Road, Suite 100, Rancho Cordova, CA
Consultant/Contractor PM:	Spencer Archer
Phone:	(970) 728-1069 (Telluride project office)
Email Report/EDD To:	spencer.archer@amec.com
Invoice To:	BP/ARC ___ Contractor x

Report Type & QC Level
Standard ___
Full Data Package ___

Lab No.	Sample Description	Date	Time	Soil / Solid	Water / Liquid	Air / Vapor		Total Number of Containers	Unpreserved	H_2SO_4	HNO_3	HCl		Comments
3	BIOFILM STANDPIPE 130919	9/19/13	1055											
4	BIOFILM ROCK 130919	9/19/13	1110											
5	BLACK SEDIMENT 130919	9/19/13	1150											
1	POND 9 FILTRATE 13091901	9/19/13	1240											1 FILTER
2	POND 9 FILTRATE 13091902	9/19/13	1330											4 FILTERS
-	0.45 MICRON FILTER 130919	9/19/13	1405											BLANK FILTER
-	COFFEE FILTER 130919	9/19/13	1410											BLANK FILTER

Comments note: If sample not collected, indicate "No Sample" in comments and single-strike out and initial any preprinted sample description.

Sampler's Name: ABBY CARTER
Sampler's Company: AMEC
Shipment Method: UPS Ship Date: 9/23/13
Shipment Tracking No: 1Z 9AE 581 03 9889 8533

Relinquished By / Affiliation	Date	Time	Accepted By / Affiliation	Date	Time
[illegible signature] / AMEC	9/23/13	1600	RYB	9-24	2:00
RYB	10-15-13	12:30	Terry Moore	10-15-13	12:30

Special Instructions:

THIS LINE - LAB USE ONLY: Custody Seals In Place: Yes / No | Temp Blank: Yes / No | Cooler Temp on Receipt: ______ °F/C | Trip Blank: Yes / No | MS/MSD Sample Submitted: Yes / No